The effect of grape temperature on the phenolic extraction and sensory perception of Méthode Cap Classique wines

By

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Summary

The first sparkling wine in South Africa was released in 1971. The South African Cap Classique Producers Association (CCPA), formed for the appreciation of Méthode Cap Classique (MCC) traditional style sparkling wines (TSW), was established in 1992 and has since contributed to the growth of these wines on a competitive footing with the international market. Generally, studies on TSW have focused primarily on the foam capability, volatile composition and autolytic character of the wines and very little on phenolic content of the wines. Phenolic compounds are important quality indicators of wine. Their composition in wine is determined by various factors including grape variety, terroir, viticultural practice, and oenological practices. In this project, MCC wines were made by the traditional method using Chardonnay and Pinot noir grapes harvested from two regions (Robertson and Darling) and stored at 0, 10, 25 and 30ºC, over two vintages (2014 and 2015). The phenolic concentration of the wine samples throughout the winemaking process was analysed by spectrophotometer and the aroma and taste of the final 9 month old sparkling wines performed. The study was aimed at investigating the effect of the grape storage temperature on the phenolic content and the sensory properties of MCCs through a quantitative phenolic analysis. The study found that MCCs made from grapes stored at lower temperatures (0 and 10ºC) had lower total phenolic content, colour intensity and total hydroxycinnamates than wines made from grapes stored at higher temperatures (25 and 30ºC) showing that there was greater phenolic extraction from grapes stored at 25 and 30ºC. The total phenolics, as measured by spectrophotometer, was below the range cited in literature for Champagne made from the same cultivars. The sensory evaluation of the MCCs comprised a sorting analysis similar to that used for beers. Separating the aroma and taste sorting of the MCCs, the study showed a grouping of the MCCs according to temperature treatments for both vintages. There were, however, clear vintage differences in terms of the attributes cited and the frequency of citations. Based on frequency of citation, 2014 MCCs made from grapes stored at 0 and 10°C were described by judges as being fruity, fresh and crisp whilst those made from grapes stored at 25 and 30°C were described as having oxidised fruit, volatile acidity and solvent-like aromas. The judges perceived less oxidation and VA (in terms of the frequency of citation) in the aroma of 2015 MCCs, although higher temperature treatments were still associated with less desirable attributes compared to lower temperature treatments. Judges were better able to separate the Darling wines according to treatments compared to the Robertson wines. This study has shown that the grape storage temperature has an effect on the phenolic extraction and the sensory perception of MCCs aged 9-months with no changes in the phenolic content observed throughout winemaking.

Opsomming

Die eerste vonkelwyn was vrygestel in 1971 in Suid-Afrika. Die “South African Cap Classique Producers Association” (CCPA) was gestig in 1992 vir die waardering van Méthode Cap Classique (MCC) tradisionele styl vonkelwyne (TSW), en het sedertdien bygedra tot die groei van hierdie wyne op 'n kompeteerende steunpunt met die internasionale mark. In die algemeen fokus studies wat gedoen is met TSW hoofsaaklik op die skuim vermoë, vlugtige samestelling en outoliese karakter van wyne en baie min op die fenoliese inhoud daarvan. Fenoliese verbindings is betekenisvol gehalte aanwysers van wyn. Die samestelling daarvan in wyn word bepaal deur ‘n verskeidenheid van faktore, insluitend druif variëteit, terroir, wingerdbou en wyn-kundige praktyke. In hierdie projek word MCC wyne gemaak volgens die tradisionele metode en strek oor twee oes jare (2014 en 2015). Chardonnay en Pinot Noir geoesde druiwe vanaf twee streke (Robertson en Darling) word gebruik en onderskeidelik gestoor by 0, 10, 25 en 30ºC. Die fenoliese konsentrasie van die wyn monsters deurgaans die hele wynmaak proses was geanaliseer met ‘n spektrofotometer en die aroma en smaak van die finale 9- maand oud vonkelwyne was uitgevoer. Die studie se hoof doelwit is om die uitwerking van die druif stoor temperatuur op die fenoliese inhoud en die sensoriese eienskappe van MCC te ondersoek met behulp van kwantitatiewe fenoliese analise. Die studie het bevind dat MCCs wat gemaak is van druiwe wat teen laer temperature (0 en 10ºC) gestoor was, het laer totale fenoliese inhoud, kleur intensiteit en totale hydroxycinnamates as die wyn van druiwe wat gestoor is by hoër temperature (25 en 30ºC). Dit toon dat daar beter fenoliese ekstraksie vanuit druiwe wat gestoor word by 25 en 30ºC verkry word. Die totale fenole, soos gemeet deur ‘n spektrofotometer, was benede die reeks aangehaal in literatuur vir Champagne wat gemaak is van dieselfde kultivars. Die sensoriese evaluering van die MCCs bestaan uit die ontleding van sortering, soortgelyk aan dié wat gebruik word vir bier. Die studie toon groepeering van die MCC’s volgens die temperatuur behandelinge vir beide oesjare met die skeiding van die aroma en smaak sortering van die MCCs. Daar was egter duidelik verskille in oesjaar in terme van die aangehaalde eienskappe en die aanhalings se herhalendheid. Op grond van die aanhaling se herhaling, die 2014 MCCs gemaak van druiwe wat gestoor is by 0 en 10ºC word beskryf deur die beoordeelaars as vrugtige, vars en fris, terwyl MCCs gemaak van druiwe gestoor by 25 en 30ºC beskryf word as geoksideerde vrugte, vlugtige suur (VA) en oplosmiddel-agtige geure. Die beoordelaars het minder oksidasie en VA (in terme van die herhaling van aanhaling) in die aroma van 2015 MCCs waargeneem, hoewel hoër temperatuur behandelings verbonde is met minder gewensde eienskappe in vergelyking met 'n laer temperatuur behandelings. Beoordeelaars was beter in staat om die Darling-wyne te skei volgens behandelings as met die Robertson-wyne. Hierdie studie toon dat die stoor temperatuur van druiwe 'n uitwerking het op die fenoliese ekstraksie en die sensoriese persepsie van 9-maand oud MCCs en geen verandering in die fenoliese inhoud was waargeneem deurgaans die hele wynmaak proses nie.

This thesis is dedicated to

My mother, Mme Florence Kelapile Mafata (1957 - 2008) for instilling free-thinking and self-discipline in me from a young age. May her soul rest in peace. My university mentor, Professor Roger Hunter, for showing me that there is a place for the unorthodox free-thinker.

Biographical sketch

Mpho Mafata was born in the town of Mogwase in the North West province on 14 August 1990. She matriculated at JM. Ntsime High school, in the same town, in 2008. In 2009 she enrolled at the University of Cape Town and obtained her B.Sc (Chemistry) degree in 2012 and B.Sc (Chemistry) Honours in 2013. She then enrolled for her MSc in Wine Biotechnology in 2014 at the Institute for Wine Biotechnology at the University of Stellenbosch whilst under the employment of the Agricultural Research Council.

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Preface

This thesis is presented as a compilation of 5 chapters. Each chapter is introduced separately and is written according to the style of the South African Journal of Enology and Viticulture.

Chapter 1	**General Introduction and project aims**
Chapter 2	**Literature review** Phenolic composition and sensory perception of traditional style sparkling wines
Chapter 3	**Research results** The effect of grape temperature on the phenolic extraction and evolution of Méthode Cap Classique wines throughout winemaking
Chapter 4	**Research results** The effect of grape temperature on the sensory perception of Méthode Cap Classique wines
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Abbreviations

ACS	American Chemical Society
ANOVA	Analysis of variance
BW	Base wine
BWpCS	Base wine post cold stabilization
CAE	Caffeic acid equivalents
CI	Colour intensity
DA	Discriminant analysis
DAD	Diode array detector
DAP	Diammonium phosphate
EE	Epicatechin equivalents
FC	Folin-Ciocalteu
GAE	Gallic acid equivalents
HPLC	High pressure liquid chromatography
IOC	Institut Oenologique de Champagne
MCC	Méthode Cap Classique
MCP	Methylcellulose precipitable tannin assay
mDP	Mean degree of polymerization
MLF	Malolactic fermentation
PCA	Principal component analysis
PVPP	Polyvinylpolypyrrolidone
QE	Quercetin equivalents
RP-HPLC	Reverse-phase high pressure liquid chromatography
SW	Sparkling wine
T2M	Wine after 2 months in the bottle
T9M	Wine after 9 months in the bottle
TA	Titratable acidity
TP	Total phenolics
TSW	Traditional Sparkling Wine
VA	Volatile acidity

Chapter 1

General introduction and project aims

1.1 Background and Introduction

The first sparkling wine of South African origin was made in 1971 from Chenin Blanc grapes and named “Kaapse Vonkel”. It was produced by the Champenoise method and paved the way for sparkling wine production in South Africa. Due to the ban of the use of the term Champagne for bottle-fermented sparkling wine, the sparkling wine was renamed Méthode Cap Classique (MCC) and the MCC association of South Africa initiated in 1992. The newly named MCC or Cap Classique uses the traditional method of production but using Chardonnay, Pinot noir and Pinot Meunier grapes (Newton, 2010).

The growth of South African sparkling wine has been steady increasing in terms of consumption, sales and exports over the past decade (SAWIS, 2016). The quality of Cap Classique wines and winemaking has increased competitively with international standards.
Internationally, research focus has been directed towards viticultural developments of grape cultivars bound for sparkling wine production and training of sensory panels to evaluate these wines (Jones *et al.*, 2014). In terms of sensory evaluation, the focus has mainly been on the physical properties (bubble quality and foaming properties) and autolytic character of the sparkling wines (Hidalgo *et al*., 2004).

There has been little focus on the phenolic composition and phenolic evolution of sparkling wines throughout winemaking and ageing. Phenolic compounds contribute to the colour, taste and mouthfeel of wines and are hence considered important quality indicators. Previous studies have shown that the phenolic content of Champagne and cava is similar to that of white wines and is dictated by the grape variety (Ibern-Gómez *et al*., 2000; Chamka *et al*., 2003). Martínez-Lapuente *et al*., 2013 investigated the total phenolic content in Spanish sparkling wines with different grape varieties and found that there was a decrease in the total phenolics. One study investigated sparkling wines of different grape varieties and showed that those with higher total phenolic content had better foam quality and were fruitier (Martínez-Lapuente *et al*., 2013). Techniques used for red table wine winemaking such as punch-downs, pump-overs, thermovinification and cold maceration to extract as much phenolics as desired (Sacchi *et al*., 2005; Bautista-Ortín *et al*., 2007) are inappropriate for TSW. Méthode Cap Classique winemaking uses free-run juice with no possibilities for maceration, hence other techniques to extract phenolic compounds from grapes need to be considered. Desired chemical attributes for TSW include low phenolics. Phenolics are linked to bitterness and astringency, oxidation reactions, and reduced aging capacity for TSW (Zoecklein, 2002) as well as browning (Ibern-Gómez *et al.,* 2000).Since for the second fermentation the conditions in the bottle are highly reductive, phenolics and SO_2 management is of utmost importance.

1.2 Aims and Objectives

The aim of this project is to evaluate the effect of grape storage temperature on phenolic extraction and sensory profile (aroma and taste) of MCC wines and to further investigate the evolution of phenolics throughout winemaking. In order to accomplish this, the objectives were as follows:

1. Make MCC using Chardonnay and Pinot Noir grapes stored at different temperatures.
2. Investigate the evolution of phenolics through-out the winemaking.
3. Evaluate the sensory profile of the resulting MCC wines.

Chardonnay and Pinot noir grapes were stored overnight at 0, 10, 25 and 30°C and pressed the next day whilst maintaining the set temperatures. The MCCs were made from blends of the two cultivars according to the traditional method with the second alcoholic fermentation, in the bottle. Spectrophotometry was used to monitor the evolution of phenolics throughout winemaking. Aroma and taste evaluation was performed on final 9-months old MCC wines using free sorting method.

There is a gap in scientific research on MCC wines compared to TSW viticulture and chemistry. The findings of this project can provide sound scientific data on MCC relevant to both researchers and South African wine industry. Producers would have scientific support to some of the decisions that need to be made at a crucial stage of MCC winemaking, at processing.

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Chapter 2

Literature review

Phenolic composition and sensory perception of traditional style sparkling wine

2.1 Introduction

Traditional sparkling wine (TSW) vinification can be comparable to white wine vinification due to the light pressing of the skins, minimal skin contact and little to no maceration. There is very little extraction of colour compounds, and generally, very little phenolic content is expected and desired. Since phenolics are linked to bitterness, astringency, reduced aging capacity (Zoecklein, 2002) and browning in TSW (Ibern-Gómez *et al*., 2000), they are kept low throughout winemaking. Studies in French Champagne, Italian Prosecco and Spanish Cava sparkling wines have shown that there is very little phenolic content in these wines (Ibern-Gómez *et al*., 2000; Chamka *et al*., 2003). The primary focus of research on TSW has been on the impact of viticultural practices, alternative grape cultivars and lees contact time/aging on lees on the sensory perception of sparkling wines (Zoecklein, 2002; Jones *et al*., 2014; Stefenon *et al*., 2014). Some studies have hence focused on the volatile content and on enhancing the sensory composition on TSW. Since most of the flavour and aroma in sparkling wine comes from the second fermentation and aging in the bottle, yeast autolysis was investigated (Alexandre & Guilloux-Benatier, 2006).

Temperature treatment and longer skin contact can increase the extraction of phenolic compounds from grape skins (Bautista-Ortín *et al*., 2005; Sacchi *et al.,* 2005). Temperature greatly affects the extraction of colour compounds from grapes (Bautista-Ortín *et al*., 2005). Winemaking treatments such as cold maceration and thermovinification are employed for the specific extraction of desired colour and taste compounds from grapes (Bautista-Ortín *et al*., 2005). Winemakers use these techniques to give red wines a richer colour and fuller body. These types of procedures are commonly used in red winemaking and less so in white or rosé winemaking. It has been shown that these desired wine attributes (intense red colour and full body) are due to the polyphenolic class of compounds. Apart from extraction of grape derived polyphenols, winemakers also use oak as chips or barrels to allow for the diffusion of oak-derived polyphenols (tannins) into wine. Not all wines can be treated the same. Special consideration has to be given to the cultivar, the particular vintage and tastings have to be performed at all stages of vinification to ensure that the desirable characteristics are maintained. Changes in temperature (storage or fermentation) can affect the chemical composition of wine (Recamales *et al.,* 2006; Del Caro *et al.,* 2014). Containers used for storage and fermentations (steel canisters, steel drums, oak barrels or glass bottles) have to be monitored to make certain that wine does not become oxidised.

The consensus is that higher temperature treatments of grapes and grape must will result in greater extraction of phenolics. In still wines, increased phenolic content may result in greater mouthfeel: wines become more astringent with a fully body and greater bitterness. In white wines, because the phenolic content is low, little contribution to the taste and mouthfeel is expected. This review will focus on SW phenolics and how the production process influences them. The sensorial perception and evaluation of SW will also be addressed.

2.2 Méthode Cap Classique (MCC) winemaking and implications for phenolic extraction

2.2.1 Overview of the traditional sparkling winemaking process

Sparkling wine made in the traditional method is predominantly produced from the cultivars Chardonnay, Pinot noir and Pinot meunier in France (Champagne and *crémant*), California, Australia, and South Africa (Cap Classique). The Spanish, Italians and Germans use native grape varieties to produce their sparkling wines. The traditional method of sparkling winemaking is referred to as *méthode traditionnelle* and *méthode classique* in French regions aside from Champagne, *méthode champenoise* in the Champagne region of France, *metodo classico* in Italy, and *méthode Cap Classique* in South Africa. These methods differ in the minimum required time of lees aging, which is generally nine-months, which is dictated by the OIV and respective national legislature (Zoecklein, 2002). The distinguishing feature about sparkling wines is the second alcoholic fermentation which creates the desired bubbles in the wine. The second fermentation distinguishes traditional method sparkling winemaking (fermentation in the bottle) from the *Charmat* method (fermentation in tanks) and carbonated sparkling wine whereby CO_2 is bubbled into the base wines (Zoecklein, 2002; Anderson *et al*., 2008; Martinez-Lapuente *et al*., 2013).

A schematic of the TSW winemaking process is presented in Figure 2.1. The grapes are harvested early, at a low berry sugar content of 17 to 20º Balling and high acidity. Grapes are usually whole-bunch pressed at low pressure (≤1.5 bars), retrieving the free-run juice which is then fermented at between 12 and 15ºC resulting in the base wines. The base wines are clarified and blended. The *liqueur de tirage* is added to sweetened base wine (20 to 24 g/L sugar) and immediately bottled for the second fermentation. The *liqueur de tirage* is a mixture of rehydrated yeast, sugar and base wine incubated at 14ºC with periodic sugar addition and aeration. Second fermentation in the bottle proceeds for 4 to 8 weeks at between 14 to 18°C. Traditional style sparkling wines have to be riddled and disgorged off the lees in the same bottle that they are sold in. Riddling requires the rotation of individual bottles around their central axis at an incline of 45º, allowing the lees to collect at the mouth of the bottle. Traditional style sparkling wine is aged for at least nine months or longer on the lees (OIV, 2016). Disgorging entails freezing of the collected lees in glycol solution and removing it as a pallet followed by immediate corking or recapping of the bottle. *Charmat* method and fortified sparkling wines do not require riddling or disgorging. Aging of *Charmat* style sparkling wine is for a few days to some weeks, fortified sparkling wines require no aging process. The two are optimised to meet consumer demands by allowing for the production of bigger volumes and cutting out the riddling and disgorging processes. The dosage is added to the dry wine in order to give the wine more flavour and sweeten it to the winemakers' preference. Brut sparkling wines (dry wines) require no dosage post disgorging (Zoecklein, 2002).

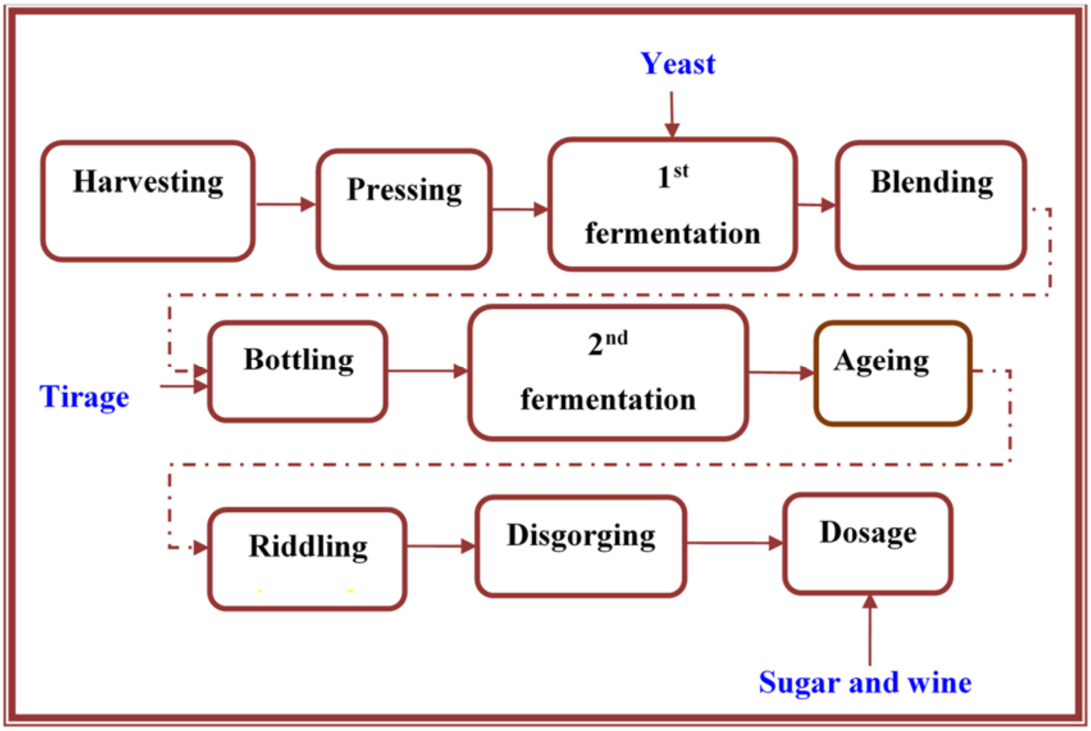

Figure 2.1: A flow diagram of the stages of the sparkling winemaking process. The aging period on the lees is dictated by national legislature. A dosage containing sugar and wine is added lastly, for taste.

2.2.2 Factors influencing the phenolic content of sparkling wine

Viticultural practices such as vine spacing, pruning and other canopy management techniques which influence the amount of sunlight exposure to the berries have been linked to changes in phenolic concentration (Smart, 1984; Jackson & Lombard, 1993). Soil, climate and irrigation influence the berry yield, but the distribution of phenolics throughout the berry is essentially the same while the concentration of the phenolics is altered (Pozo-Bayón *et al*., 2004). Different clones of Chardonnay and Pinot noir have different berry size and maturation rates hence yield different concentrations of phenolics at different sugar concentrations. Red cultivars have more anthocyanins than white cultivars, which in turn have a higher concentration of phenolic and hydroxycinnamic acids (Chamkha *et al*., 2003; Bautista-Ortín, *et al*., 2007; Mikes *et al*. 2008). The choice for a white and red cultivar to be used for TSW elaboration is based on different criteria. For example, for a white cultivar, TSW winemakers choose Chardonnay clones based on the desired sugar:acid balance, as the level of acidity and sugar have to be appropriate for a double fermentation, while for red cultivars, the choice of Pinot noir is based on their fruity notes (Zoecklein, 2000). However, according to our knowledge no studies have investigated the effect of grape temperature on the phenolic composition of sparkling wines.

2.3 Phenolic composition and chemical analysis of sparkling wine

2.3.1 Chemical analysis of sparkling wine

For the analysis of phenolics, studies used spectrophotometric methods similar to those that have been used in still wines with minor alterations such as degassing of samples (Somers & Evans, 1977; Iland *et al*., 2000). Although some studies have used the Folin-Ciocalteu (FC) method of phenolic analysis, recent studies used adaptations of Somers and Evans (1977) to analyse TSW phenolics. The (FC) method measures the total reducing/anti-oxidant capacity of a sample (Singleton, 1999; Waterhouse, 2001). The FC method is best for red wines because they have a high concentration of phenolic compounds. White wines have low phenolic concentration, hence the measurements become invalid when taking into account interferences (Singleton, 1999; Waterhouse, 2001). Sample preparation with FC reagent is more selective but the sample preparation is tedious and the reagent is costly (Waterhouse, 2001). The Somers & Evans (1977) method was developed for red wine analysis and later adapted to fit white wine matrix (Somers & Ziemelis, 1985). Studies on TSW analysis adapted the Somers & Ziemelis (1985) method with good consistency in results investigating overall changes in phenolic content (Ibern-Gómez *et al*., 2000; Chamkha *et al*., 2003).

2.3.2 Phenolic composition of sparkling wine

The grape cultivar, clone, viticultural practices and vinification all affect the composition and concentration of phenolic compounds. The grape berry phenolic composition and concentration are good indicators of what ultimately goes into wine. Traditional style sparkling winemakers do not desire a high phenolic content, therefore they harvest early when the phenolic maturity is low and press lightly so as to obtain free-run juice with low levels of phenolics. Gentle pressing of these grapes results in even lower phenolic concentrations in the juice. Thus TSW have lower phenolic concentration compared to table wines (Chamkha *et al*., 2003).

Grape-derived phenolic compounds can be categorized into two main groups, namely non-flavonoids (hydroxybenzoic/phenolic acids and hydroxycinnamic acids) with lower molecular weight and flavonoids (anthocyanins, flavan-3-ols and tannins) with higher molecular weight and a typical common C6-C3-C6 molecular structure (Fernandéz de Simon *et al*. 1992; Pozo-Bayón *et al*., 2003; Monagas, Bartolomé & Gómez-Cordovés, 2005). Flavonoids are compounds located mostly in the skin and seed of the berry while non-flavonoids are located throughout the berry, but are more concentrated in the flesh (Perez-Coello & Díaz, 2009; Ribéreau-Gayon *et al*., 2006). Non-flavanoids are extracted into the juice upon pressing, flavonoids are extracted to a lesser extent and hence winemakers employ several techniques such as maceration and thermovinification to encourage the extraction of flavonoids from the skins and seeds if desired (Perez-Coello & Díaz, 2009; Ribéreau-Gayon *et al*., 2006). Due to these viticultural and vinification practices, the phenolic content of sparkling wines comprises mainly non-flavonoids and low flavonoid concentration (Ibern-Gómez *et al*, 2000; Andrés-Lacueva *et al*.,1996).

Studies have shown that the total phenolic content of TSW are within the range reported for white wines (*i.e.* 50-350 mg/L GAE) with hydroxycinnamic acids being the major component (Cheynier *et al*., 2008; Ibern-Gómez *et al*., 2000; Pozo-Bayón *et al*., 2003a). Studies investigating the evolution of these phenolic compounds in Chardonnay and Pinot noir throughout TSW winemaking have found that no change in the levels occurs. Those that investigated other grape cultivars besides Chardonnay and Pinot noir showed that the wines had greater phenolic concentrations (200 – 500 mg/L) that decreased throughout TSW winemaking (Pozo-Bayón *et al*., 2003a). The decrease of anthocyanins during cold-stabilization was attributed to the adhering of phenolic compounds to fining agents, bentonite and Polyvinylpolypyrrolidone (PVPP) (Mazauric and Salmon, 2005; Martínez-Lapuente *et al*., 2013). Once the TSW is bottled, the phenolic concentrations fluctuate over the period of aging on lees with no statistically significant increase or decrease. This has been attributed to their initial adsorption to yeast cells during the first few months of aging and subsequent release during yeast autolysis (Mazauric and Salmon, 2005). Some studies found a decrease in total phenolics from base wines and across 9-month aging (Martínez-Lapuente *et al*., 2013; Stefenon *et al*., 2014) whilst another study found an overall lack of change in phenolics from base wines across 9-months aging (Gil-Muñoz *et al*., 1999). Browning (measured at 420 nm) of TSW was shown to increase after 15-months on the lees due mainly to the presence of hydroxycinnamic acids (Ibern-Gómez *et al*, 2000).

2.4 Sensory evaluation and sensory perception of sparkling wines

2.4.1 Sensory evaluation of sparkling wine

Sparkling wine sensory evaluation is very different from table wines due to the effervescent nature of the wines. The bottle pressure has been shown to have little impact on the foam quality and aroma intensity but a link between the chemical composition (not including phenolics) and foam properties has been made (Pueyo *et al.*, 1995). The time between tasting and pouring has to be minimised, as it has been shown to have an impact on the sensory perception of sparkling wine. Panel uniformity in terms of an equal amount of wine poured, randomization and time between tasting and pouring needs to be ensured as far as possible (Hood-White & Heymann, 2015).

Sensory evaluation studies performed on TSW have mostly employed descriptive analysis (DA). DA is a very useful sensory analysis tool in terms of the amount of quantitative and qualitative data that may be generated. It allows for the generation of sensory descriptors along with their intensities. DA can become costly, time consuming, is labour-intensive, it usually requires tasters to be trained and only a few wines may be tasted at a time. DA is usually used when there are a few wines to be analysed and can become very exhausting for tasters when many wines are presented to them (Polidori *et al*., 2009; Hidalgo, *et al*., 2004; Vannier *et al*., 1999).

Three-way sorting exercises have been used to evaluate the differences between wines. The exercise is useful in generating statistical information on these differences. It also has the disadvantage that very few wines can be analysed at a time due to the many 3-way permutations, training is often necessary, it can become exhausting to judges and quantitative data (intensities of attributes) is lost. Three-way sorting also carries some monetary and time costs.

Directed and free sorting analyses can be used to overcome the short-falls of both the DA and 3-way sorting. It is best used for gathering quantitative data for similarity grouping, but can be used to gain qualitative data on the attributes of wines with minimal training. Untrained and trained panels have previously been shown to produce similar results (Parr, *et al*., 2010; Lelièvre *et al*., 2008; Chollet, *et al*., 2011). Some studies have shown a difference in results when it comes to expertise (Ballester *et al*., 2008; Chollet & Valentin, 2001; Patris *et al*., 2007). Sorting requires simultaneous analysis of all wines investigated; hence total amount of wines cannot be divided over several sessions as is often done in DA. Sorting analyses are dependent on the type of product assessed and the number of wines assessed has an impact on the effectiveness of the panel (Chollet *et al.,*2014). Effervescence adds to panel fatigue. Based on studies done on beer and wine, Chollet *et al.,* (2014) advised that between 9 and 20 products be assessed in one sitting with 12 being the optimum number (Chollet *et al*., 2014).

The exercise is often split between the aroma and taste sorting profile, to minimize panel fatigue. The panel may be given a collection of attributes during a sorting which works to combat panel fatigue and increases the number of wines which may be analysed. It has also been shown that the more similar the wines the more difficult the exercise becomes (Ballester, 2013; Chollet, *et al*., 2014; Valentin *et al*., 2016). The exercise requires more than 20 judges to give statistically sound results (Faye, 2004; Lelièvre *et al*., 2008; Chollet *et al*., 2011). The difficulty with sensory evaluation of Cap Classique wines is the different vocabulary that judges use to communicate the attributes they perceive. In cases such as this, a free-sorting allows for the unrestricted generation of attributes from judges which can be narrowed down by a panel vocal discussion to reach consensus on similar attributes and grouping of attributes (Chollet *et al*., 2014).

2.4.2 Sensory perception of sparkling wine

When it comes to sensorial aspects of sparkling wines, the most important and iconic attribute is the effervescence. The foam properties affect the perception of aroma and mouthfeel. The pH, organic acids, proteins and acids affect both the formation and time-stability of foam in sparkling wines. Proteins and acids had a positive effect on the formation of foam. Low acidity, proteins and amino acids had a negative effect on the time-stability of foam (Maujean *et al*., 1990; Andrés-Lacueva *et al*., 1996). The foaming capability of TSW was found to positively correlate to greater concentrations of alcohol, total acidity, fructose, proteins and glutamine compared to wines with lower concentrations of these compounds. The foaming capability, however, inversely correlates to greater concentrations of glucose and lactic acid. Wines that undergo MLF will hence experience lower foaming ability (Andrés-Lacueva *et al*., 1996).

The aroma of sparkling wine is also very important and many studies focus more on the olfactory attributes. Studies investigate attributes such as olfactory intensity, fruitiness (exotic and citrus fruits), varietal aromas, floral, vegetal, yeasty, mould, reductive and oxidized notes from young TSW (Pérez-Magarino *et al*., 2013) whilst attributes such as toasty, buttery, caramel, butterscotch are more sought after in sparkling wines aged older than 9-months (Francioli *et al.*, 2003). In terms of the aging of TSW, ethyl lactate (cheesy) and diethyl succinate (fruity/ sugary/ floral scents) have been found to fluctuate with aging and are good markers for the age of sparkling wine (Ribéreau-Gayon, 2006; Pueyo *et al*., 1995; Francioli *et al*., 2003).

Vanilla sensory attribute of sparkling wines develops during the aging of the wine on lees. The vanilla intensity is less in the base wine compared to the finished sparkling wine. White grape varieties (Chardonnay and Pinot Blanc) used for the elaboration of sparkling wine have more vanilla intensity than red grape varieties, Pinot noir and Pinot Meunier (De la Presa-Owens *et al*., 1998).

Compounds responsible for SW aroma are derived from the grape, from fermentation and aging on lees. Grape-derived aroma attributes include floral, fruity and herbaceous. Aroma and taste attributes derived from aging on lees may include toasted bread, caramel, woody, oak (whether the SW has or has not been oaked), liquorice, yeast autolytic character and creamy notes (Vannier *et al*., 1999; Torrens *et at*., 2010; Riu-Aumatell *et al*., 2013).

Yeast autolysis is the degeneration of yeast cells that releases yeast cell products like polysaccharides, glycoproteins, lipids and nucleic acids (Feuillat *et al*., 1982; Martínez-Rodriguez *et al*., 2001; Fornairon-Bonnefond *et al*., 2002). These yeast autolysis products have a distinct sensorial character referred to as the “autolytic character” which has previously been associated with attributes such as toasty, bread, butter, and butterscotch. The proteins released during yeast autolysis have previously been connected to the perception of “fuller body’ in wines (Martínez-Rodriguez *et al*., 2002; Martínez-Rodriguez & Pueyo, 2009). Liger-Belair (2005) showed that there are some volatile compounds (aldehydes, esters, higher alcohols and lipids) that are released from the yeast cells during yeast autolysis and some volatile phenols are generated by yeast enzymatic decarboxylation of coumaric and ferulic acids. Volatile compounds identified in SW could potentially be used as age markers, discriminating between old and young wines (Francioli *et al*, 2003). Chapentier *et al*., (2005) showed a link between yeast cell derived nucleic acids, release of yeast cellular contents (during autolysis and hydrolysed intracellularly by enzymatic reactions) and SW mouthfeel and flavour.

Most phenolic compounds are responsible for mouthfeel sensations and due to their low volatility, have little to no olfactory properties. Phenolic compounds give colour, astringency, acidity/ sourness and bitterness to wine. The threshold of gustatory perception of these compounds in wine is much higher than their actual concentration in traditional style sparkling wine (Gawel, 1998). Phenolic compounds act in combination with wine pH, alcohol, SO_2 concentration and total acidity to elicit a sensorial response. TSW winemaking extracts the free-run juice containing mainly flavonols and hydroxycinnamic acids which give bitterness to wines. Tannins introduce astringency and bitterness to the wines, but given their low concentration in sparkling wines, their contribution to the mouthfeel of these wines is limited (Chamkha *et al*., 2003).

2.5 Conclusion

Research studies on traditional style sparkling wine made from Chardonnay and Pinot noir cultivars identified the major phenolic compounds to be hydroxycinnamic acids and hydroxybenzoic acids. The concentration of these compounds and the total phenolic content was similar to that measured in white and rosé wines made with no maceration and little skin contact. This low concentration of phenolic compounds in these sparkling wines is present at or below the sensorial limit of detection. The perception of sensory attributes related to these compounds (bitterness, acidity and astringency) was attributed to a combination of factors.

Given the current research on sparkling wines, there is a gap in the knowledge on the effect of temperature manipulations on grapes used for the elaboration of sparkling wine and the effects of such treatments on the phenolic extraction and sensory perception of Cap Classique wines.

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Chapter 3

The effect of grape storage temperature on the phenolic extraction and evolution in Méthode Cap Classique wines throughout winemaking

3.1 Introduction

Winemakers desire less phenolic content in traditional sparkling wine (TSW), many viticultural and vinification efforts are made to ensure this. From the early harvest to the light press and the lack of skin contact, free-run juice is obtained at low phenolic concentrations (Zoecklein, 2002). The extraction of phenolics during winemaking is subject to many factors with temperature playing a very critical role. It was shown that chilling grapes at 10ºC decreased phenolic extraction into juice (Gil-Munoz, 1999). TSW winemakers employ this strategy to make certain that the wines maintain a fresh and fruity palate (Zoecklein, 2000; Hidalgo *et al.*, 2004). In order to obtain mouthfeel characteristics associated with greater phenolic content such as a fuller body and greater astringency, TSW winemakers use extended lees contact time, malolactic fermentation, innovative mixtures for their tirage and dosage (Zoecklein, 2002).

The measurement of oenological parameters (pH, alcohol, SO_2, volatile acidity, residual sugar and total acidity) is important as these parameters can impact the sensory perception of TSW (Pérez-Magarino, *et al*., 2013). Low TA concentrations have been associated with flatness in wines and high TA associated with sourness (Zoecklein, 2002). The RS of the final wine (unless a brut) depends on the dosage (Zoecklein, 2002). Although pH and alcohol have been shown to have an impact on the sensorial perception of phenolic-related attributes such as bitterness, astringency and body, the phenolic content of TSW is below the sensory threshold and so far no impact has been shown in TSW (Gawel, 1998; Zoecklein, 2002; Chamka *et al.*, 2003).

Two studies on the progression of phenolics throughout winemaking found contradicting results. One study on Cava TSW made using Spanish cultivars showed they decreased throughout winemaking and had higher concentration of phenolics compared to Chardonnay and Pinot noir cultivars (Pozo-Bayón *et al*., 2003a). Another study on Champagne made from a blend of Chardonnay and Pinot noir grapes reported phenolic concentrations lower than those reported for Cava and additionally showed no change in phenolics throughout winemaking (Chamkha *et al.,* 2003). Studies on the phenolic content and phenolic progression of Méthode Cap Classic (MCC) wines throughout TSW winemaking have yet to be published. This study hopes to show that the temperature of grapes at pressing influences the extraction of phenolics. Additionally, the evolution of phenolics throughout winemaking was evaluated.

3.2 Materials and methods

3.2.1 Vinification and Sampling

Chardonnay and Pinot noir grapes were sourced from Graham Beck farm in Robertson and Groote Post farm in Darling for the vintages of 2014 and 2015. The grapes were harvested in the early morning and transported, on the day, to the ARC Infruitec-Nietvoorbij experimental cellar. For each region and for each cultivar (Chardonnay and Pinot noir), two tons of grapes were divided into four groups and stored in temperature specific cold rooms overnight at 0°C, 10°C, 25°C and 30°C until they acclimatized to the set temperature. Temperature probes were inserted in and between grapes to ascertain that the grapes reached and maintained the set temperature.

Each temperature group was divided into three repeats and the grapes whole-bunch pressed at a pressure of between 1.0 and 1.5 bar into 90 litre drums and 50 mg/L SO_2 added. The Chardonnay was treated the same as the Pinot Noir. The juice yield was 48 to 50% L/kg. The free-run juice was then sampled (Figure 3.1) and stored at 14°C and allowed to acclimatize. The must was then inoculated with 0.3 g/L *S. cerevisiae* IOC18-2007 (CDS Vintec, Stellenbosch, South Africa) yeast, 0.5 g/L diammonium phosphate (DAP) was added and the wines to at 14 °C. The wines were racked, 50 mg/L SO_2 was added and a sample of the base wine taken. The base wines were clarified using 0.75 g/L bentonite and cold stabilized at 0°C for 2 weeks, a sample was then taken (BWpCS). The base wines where racked once more. Corresponding Pinot noir and Chardonnay treatments were then blended in a 50/50 ratio and allowed to stand for a further week, samples of the blends were then taken. The blends were sweetened to 24 g/L with cane sugar, inoculated with a 4 % tirage liqueur made-up of the same yeast as the one from the first fermentation, bottled under nitrogen gas and capped with a crown capper. The second fermentation was tracked by measuring the pressure in the bottle, one bottle per treatment was sacrificed. Once the pressure stabilised, indicating the end of fermentation, a sample was then taken. The wines were shelved horizontally and allowed to mature in the bottle for a further 7 months. The wines were riddled and disgorged at Simonsig cellar, Stellenbosch, South Africa. *Liqueur d'expédition/ Liqueur de dosage* was not added, the final brut wines were recapped and some were sampled for chemical and sensory analysis. A schematic of the Cap Classique winemaking protocol and sampling is shown in Figure 3.1.

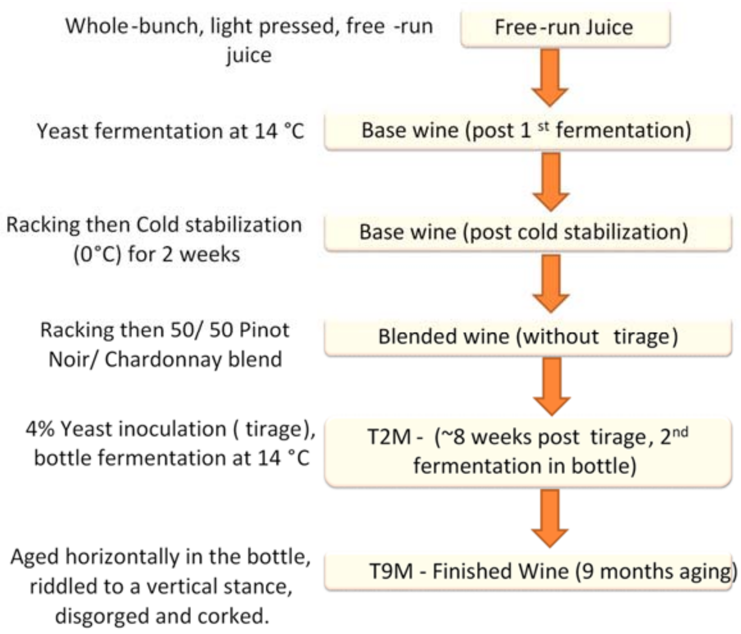

Figure 3.1. Diagram of the MCC winemaking protocol with the right pane showing the stages that were sampled for chemical analysis

3.2.2 Oenological parameters

The sugar content of the free-run juice at room temperature (after temperature treatment) was analysed using a PR-30α (alpha) digital refractometer. Wines were analysed for pH and titratable acidity (TA) on a Tim868 auto-titrator using American Chemical Society (ACS) grade reagents from Hanna Instruments (Pty) Ltd, Rhode Island, US. Free and total sulphur dioxide (SO_2) concentrations were analysed according to the Ripper method using ACS grade reagents (Vahl and Converse, 1980). The alcohol concentration was analysed on an Anton Paar alcoholizer Wine M. Residual sugar (RS) and Volatile acidity (VA) were analysed degassed samples at Koelenhof laboratorium, Stellenbosch, South Africa using Fehlings method and distillation, respectively.

3.2.3 Colorimetric analysis

The analysis was adapted from Somers and Ziemelis, 1985. All analyses were performed in triplicate. Prior to analysis, sparkling wines (T2M and T9M) were degassed. All samples were centrifuged at 13 000 rpm in 2 mL micro-centrifuge tubes for 10 minutes and the supernatant decanted. The supernatant was acidified with a 1 M HCl solution (using 32 % HCl from Sigma-Aldrich) and allowed to stand for 3 hours. The absorbance was read at 420 and 520 nm for non-acidified samples and 280 and 320 nm for acidified samples on a Multiskan GO 1510-02586 spectrophotometer. All spectral measures were converted to 10 mm path-length absorbance units. Ultrapure water was obtained using a Millipore water purification system. Quantification of total phenolics was based on standard curve of 200, 100, 50, 25 and 10 mg/L of gallic acid prepared at the same time using gallic acid (monohydrate) purchased from Sigma-Aldrich. Concentrations were expressed in mg/L gallic acid equivalents (mg/L GAE) using the absorbance of acidified samples at 280 nm. Total hydroxycinnamic acids were calculated as the absorbance at 320 nm acidified/ at low pH (A_{320} – 2.5). The colour intensity (CI) and colour hue (CH), at actual wine pH (not acidified) and SO_2 level, were calculated as follows: CI = $A_{520} + A_{420}$ and CH = A_{420} / A_{520}.

3.2.4 Statistical analysis

Multivariate analysis (principal component analysis, PCA) was performed on colorimetric data and oenological parameters using XLStat (Version 2016, Addinsoft, New York, USA) in order to find statistical relationships between temperature treatments and the measured data. Univariate analyses (analysis of variance, ANOVA) were performed using the GLM Procedure of SAS software (Version 9.4; SAS Institute Inc., Cary, USA). Fisher's least significant difference was calculated at the 5% level ($p < 0.05$) to compare treatment means.

3.3 Results and discussions

3.3.1 Vinification and Oenological parameters

Sugar measurements of the grape juice were taken at room temperature after the grapes were temperature treated. The overnight storage of grapes at 25 and 30°C resulted in lower berry sugar concentration (Tables 3.1 and 3.2) compared to grapes stored at 0 and 10°C (true for both farms and both vintages) with the exception of the Robertson 25°C treatment of 2014. The differences in berry sugar concentration may have been due to the conversion of sugar to alcohol due to the activity of native yeast during storage at higher temperatures since there was no SO_2 added before storage. The average pH of Chardonnay samples was lower than that of Pinot noir samples for both farms and during both vintages with higher temperatures having higher pH (Tables 3.1 and 3.2). This is contrary to what is expected since higher temperature should result in higher extraction of acids from the grapes and higher solubility. The TA of Darling juice was higher than that of Robertson for both vintages (Tables 3.1 and 3.2). All parameters were within the ranges reported in literature (Ganss *et al.,* 2011; Zoecklein, 2002).

Free-run juices successfully fermented to dryness for both alcoholic fermentations with the exception of the 25ºC treatments during 2015. Darling wines were irretrievably in a stuck-fermentation during the first fermentation while Robertson wines were stuck during the second fermentation. This may have been due to the uneven distribution of temperature during storage, which resulted in poor grape quality at the moment of pressing. These vinification difficulties experienced during 2015 resulted in the oxidation of the 25°C treatments.

The second fermentation in the bottle proceeded at 14ºC and was tracked by measuring the average pressure in the bottle once a week. Steady state of the pressure indicated that fermentation had proceeded to dryness, and samples were taken (T2M). The wines were then allowed to age for a further 7 months on lees and within the bottle, there was no further increase in pressure after eight weeks in the bottle. The average pressure in the bottle was 6.4 bars with no differences in the final pressure across treatments.

Table 3.1: Oenological data of 2014 juice samples for Robertson and Darling farms.

	Chardonnay				**Pinot noir**			
Robertson	**0°C**	**10°C**	**25°C**	**30°C**	**0°C**	**10°C**	**25°C**	**30°C**
pH	3.08	3.09	3.18	3.17	3.22	3.17	3.20	3.19
TA	7.34	8.81	4.15	6.77	5.52	6.56	5.43	8.53
Sugar	19.3	18.2	22.6	17.8	22.2	21.1	24.2	15.1
SO_2 (total)	6	6	6	6	6	7	11	12
SO_2 (free)	4	3	3	3	3	3	3	2
Darling	**0°C**	**10°C**	**25°C**	**30°C**	**0°C**	**10°C**	**25°C**	**30°C**
pH	3.06	3.12	3.25	3.18	3.14	3.21	3.28	3.26
TA	10.79	12.80	12.84	13.61	13.47	11.98	11.69	12.91
Sugar (°B)	17.5	18.8	16.6	16.5	18.5	17.5	16.3	15.8
SO_2 (total)	6	7	15	10	11	14	19	12
SO_2 (free)	3	3	3	3	3	2	3	3

Note: samples were taken without replicates; hence no statistical analyses were performed. TSO_2 - total sulphur dioxide; FSO_2 - free sulphur dioxide; TA - titratable acidity.

Table 3.2: Oenological data of 2015 juice samples for Robertson and Darling farms.

	Chardonnay					**Pinot noir**		
Robertson	**0**	**10**	**25**	**30**	**0**	**10**	**25**	**30**
pH	3.18c	3.22c	3.31c	3.30c	3.49b	3.81a	3.23c	3.26c
TA	9.07b	7.83cd	7.87cd	8.13c	7.13d	3.30e	10.53a	7.73cd
Sugar	19.7c	20.6a	19.67a	19.3d	20.1b	19.5cd	10.00ab	18.6e
SO_2 (free)	18ab	19a	19.57c	13ab	11ab	6b	18.07f	9ab
Darling	**0**	**10**		**30**	**0**	**10**		**30**
pH	3.20c	3.11d		3.41a	3.27b	3.20c		3.43a
TA	12.17c	9.67d		14.17b	10.97dc	10.40d		18.77a
Sugar	19a	19c		18e	20a	19ab		17e
SO_2 (free)	-	13a		13ab	9b	10ab		11ab

Note: Triplicate samples were taken at pressing after temperature treatments were done. The following are averages over the triplicates with statistical differences calculated at p<0.5 across treatments and winemaking stages. TSO_2 - total sulphur dioxide; FSO_2 - free sulphur dioxide; TA - titratable acidity.

The stages of winemaking account for over 30% of the variation in the Robertson (Figures 3.2 and 3.4) and Darling (Figure 3.3 and 3.5) oenological parameters. Both Robertson and Darling farms during both the 2014 and 2015 vintages showed significant increases in VA, alcohol and pH throughout winemaking (from the base wines before cold stabilization until 9-month old MCCs; data not shown) with the exception of the 25°C treatments from 2015 which, as previously mentioned, had significantly higher levels of the measured oenological parameters (data not shown). The increase in alcohol was proportional to the berry sugar content and the sugar addition at the second fermentation. For both vintages and both farms, there were no significant differences in the oenological parameters across treatments with the exception of the VA of higher temperature treatments being higher than lower temperature treatments. All wines were fermented to dryness hence the final MCCs were bruts with less than 8 g/L (Zoecklein, 2002).

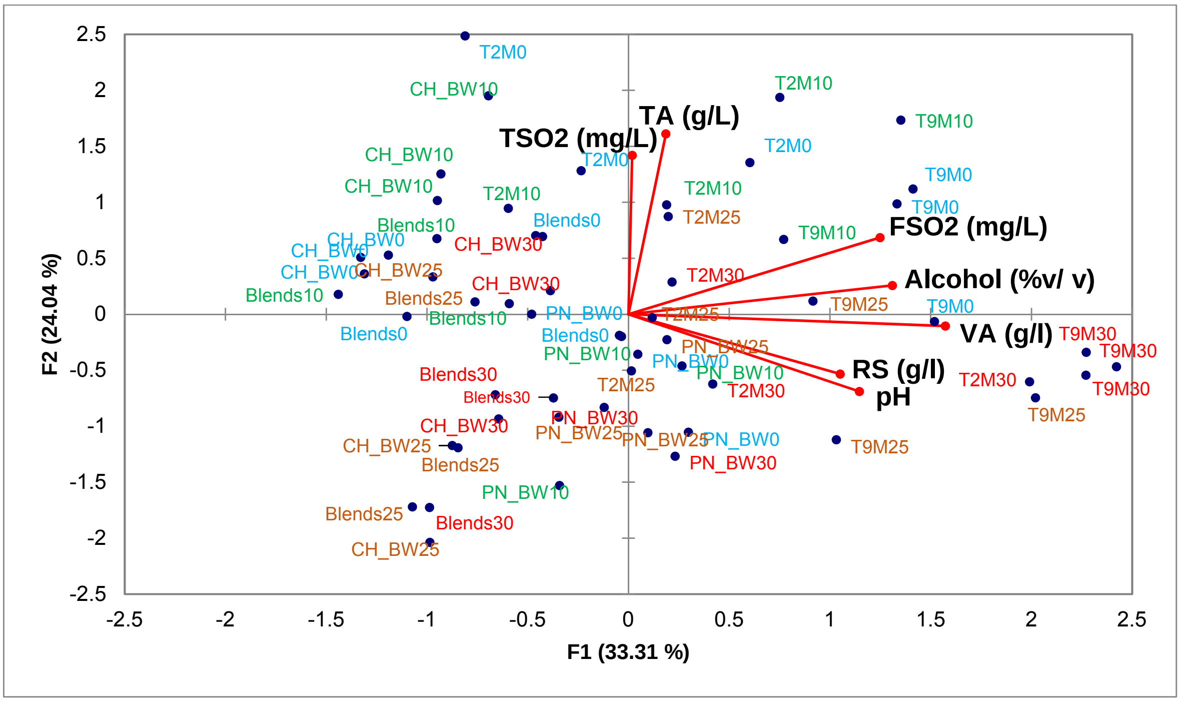

Figure 3.2. Principal component analysis (PCA) biplot of 2014 Robertson oenological parameters (total sulphur dioxide-TSO2, free sulphur dioxide-FSO2, titratable acidity-TA, volatile acidity-VA, residual sugar-RS, pH and alcohol). Wines sampled before (CH_BW and PN_BW) and after (CH_BWpCS and PN_BWpCS) cold stabilization, after second fermentation (T2M) and the final wines aged for nine months (T9M) samples

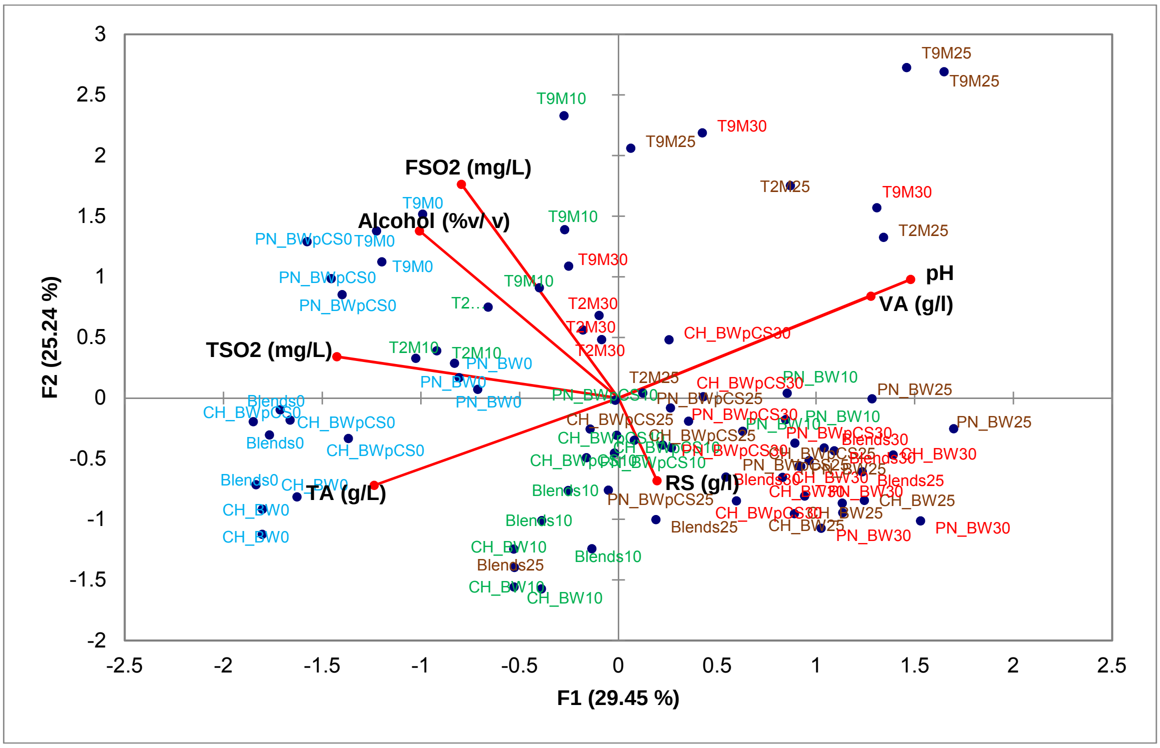

Figure 3.3. Principal component analysis (PCA) biplot of 2014 Darling oenological parameters (total sulphur dioxide-TSO2, free sulphur dioxide-FSO2, titratable acidity-TA, volatile acidity-VA, residual sugar-RS, pH and alcohol). Wines sampled before (CH_BW and PN_BW) and after (CH_BWpCS and PN_BWpCS) cold stabilization, after second fermentation (T2M) and the final wines aged for nine months (T9M) samples

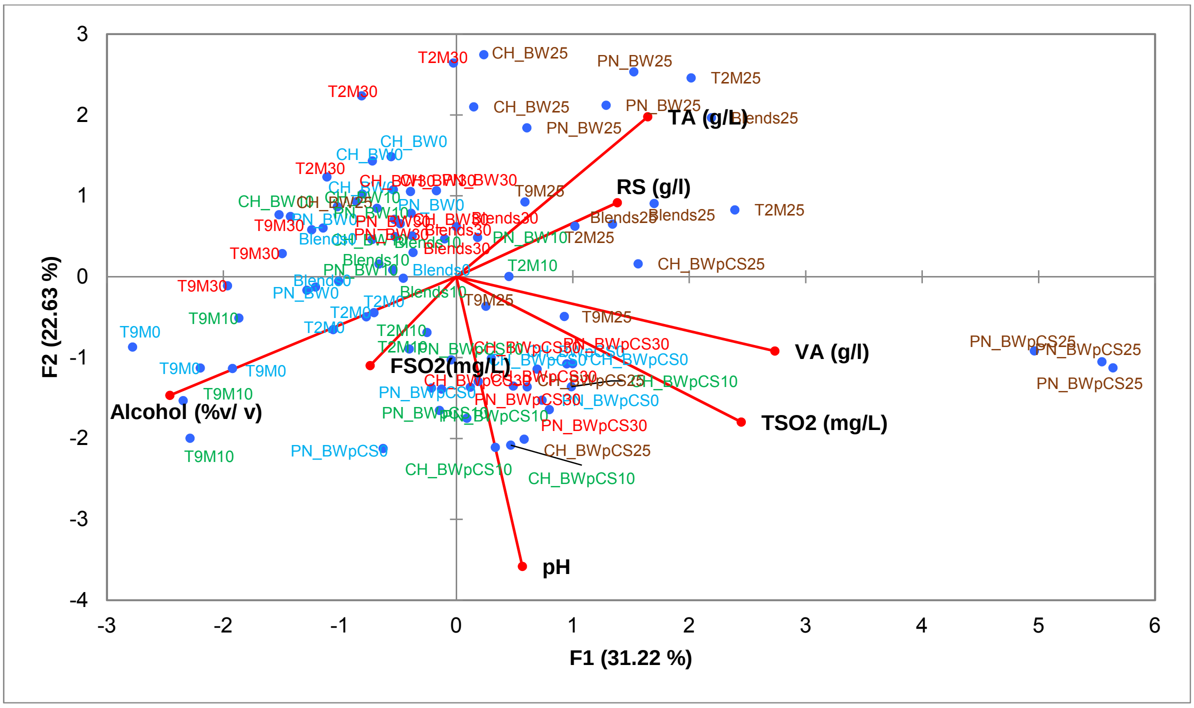

Figure 3.4. PCA biplot of 2015 Robertson oenological parameters (total sulphur dioxide-TSO_2, free sulphur dioxide-FSO_2, titratable acidity-TA, volatile acidity-VA, residual sugar-RS, pH and alcohol). Wines sampled before (CH_BW and PN_BW) and after (CH_BWpCS and PN_BWpCS) cold stabilization, after second fermentation (T2M) and the final wines aged for nine months (T9M) samples

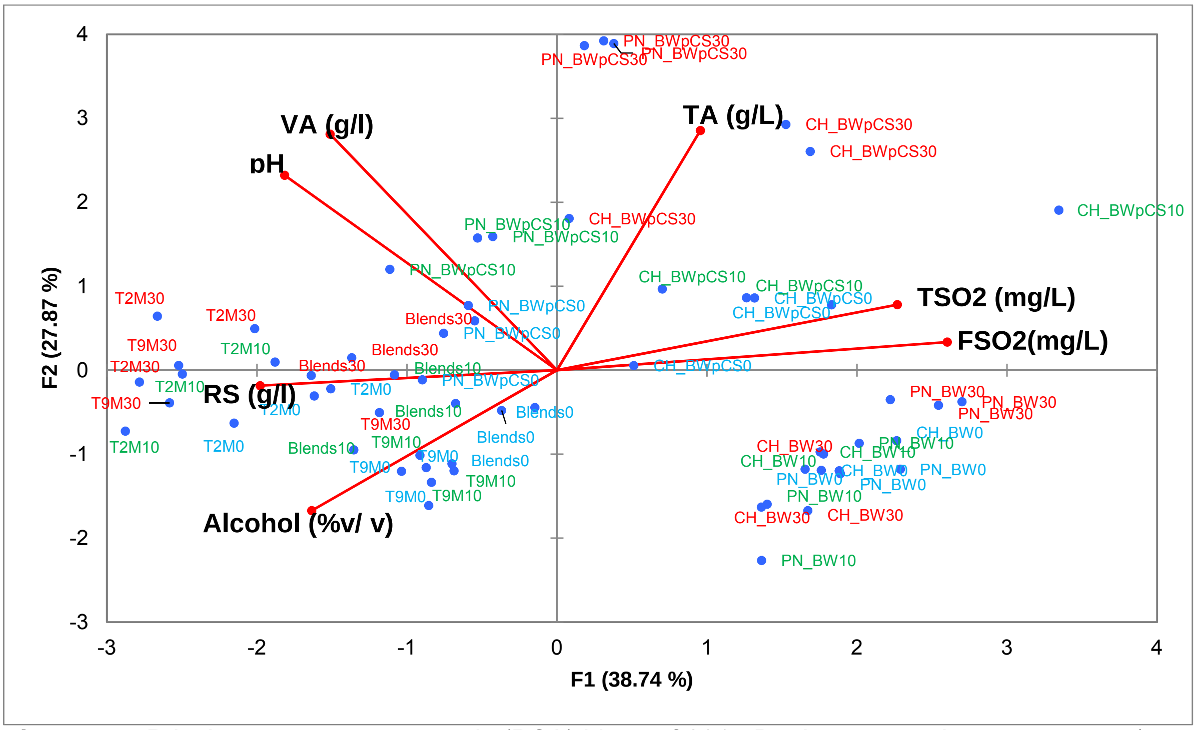

Figure 3.5. Principal component analysis (PCA) biplot of 2015 Darling oenological parameters (total sulphur dioxide-TSO_2, free sulphur dioxide-FSO_2, titratable acidity-TA, volatile acidity-VA, residual sugar-RS, pH and alcohol). Wines sampled before (CH_BW and PN_BW) and after (CH_BWpCS and PN_BWpCS) cold stabilization, after second fermentation (T2M) and the final wines aged for nine months (T9M) samples.

3.3.2 Colorimetric analysis of 2014 vintage

In order to quantify the phenolic extraction, the total phenolics (TP) were measured at 280 nm. Since the hydroxycinnamic acids were shown to be the highest contributors to the TP and play a role in the stability and evolution of TSW (Ibern-Gómez *et al.*, 2000), total hydroxycinnamic acids (TH) were measured (at 320 nm). Hue (CH) and intensity (CI) are two ways of characterising the colour properties of wines based on absorption at 420 nm and 520 nm for yellow/brown and red colour, respectively.

The major cause of variation in the colorimetric data of both Robertson (Figure 3.6, 71%) and Darling (Figure 3.7, 62%) was due to the temperature treatments. Higher temperature treatments (25ºC and 30ºC) grouped together and so did the lower temperature treatments (0ºC and 10ºC) with good repeatability between the biological repeats. Across all stages of winemaking, the higher temperature treatments were significantly higher in TP, CI and TH than the lower temperature treatments (Table 3.3). The total phenol content was lower than the 176 to 195 mg/L GAE range reported for Champagne in literature (Chamkha *et al.*, 2003). The colour hue of lower temperature treatments was lower than that of higher temperature treatments due to their low absorption at A_{520} caused by lesser phenolic extraction from Pinot noir grapes stored at lower temperatures. Similar to the study by Gil-Muñoz *et al.*, (1999), there were no significant differences seen in the phenolics across winemaking (Table 3.3). Prior to blending the Pinot noir base wines observed the same treatment patterns mentioned above but the Chardonnay samples were not affected by the treatments the same way as the other treatments (data not shown). The Chardonnay base wines were higher in CH due to their low absorption at A_{520}, white cultivars have less anthocyanins than red cultivars (Ribéreau-Gayon,1982). There were no consistent patterns observed in the Chardonnay phenolic measurements in relation to the treatments. There was a statistically significant increase in the CH from base wine blends to the final wine (T9M) implying a loss of absorption at A520 which may have been due to the adsorption of anthocyanins to yeast cell walls (Vasserot *et al.*, 1997).

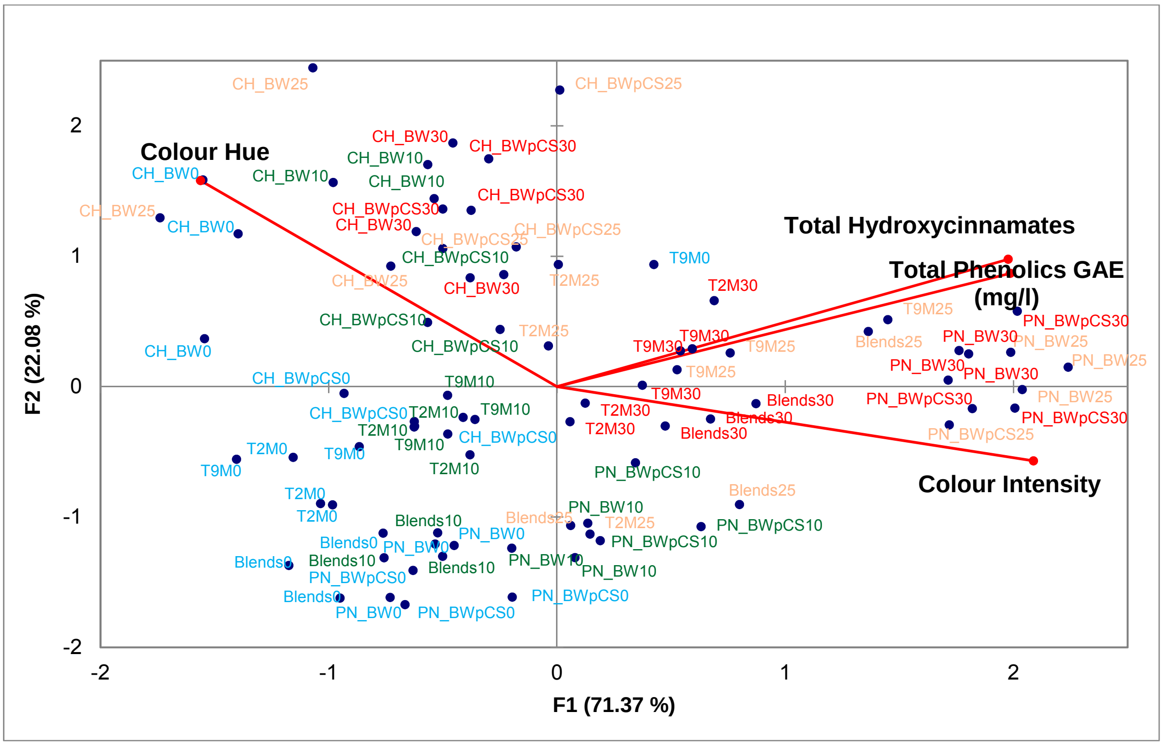

Figure 3.6: PCA biplot of 2014 Robertson colorimetric analysis (Colour hue, colour intensity, total phenolics in mg/L GAE, total hydroxycinnamates). Wines sampled before (CH_BW and PN_BW) and after (CH_BWpCS and PN_BWpCS) cold stabilization, after second fermentation (T2M) and the final wines aged for nine months (T9M) samples.

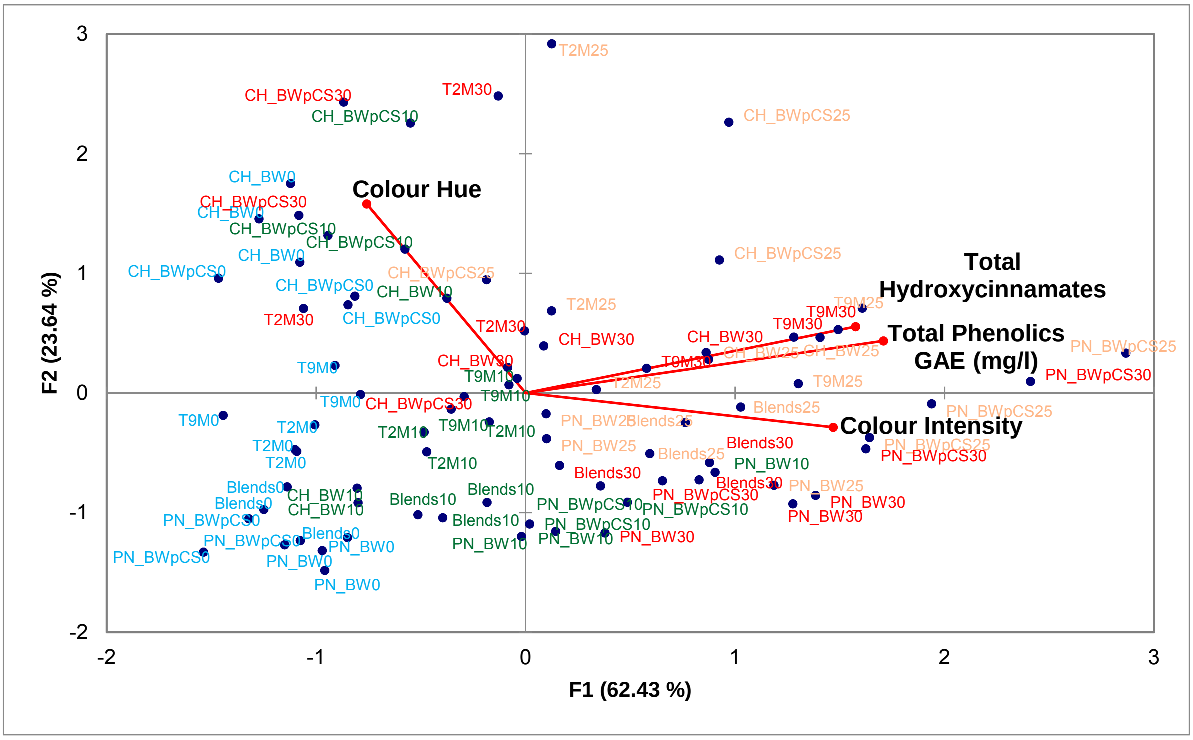

Figure 3.7: PCA biplot of Darling 2014 colorimetric analysis (Colour hue, colour intensity, total phenolics in mg/l GAE, total hydroxycinnamates) results. Wines sampled before (CH_BW and PN_BW) and after (CH_BWpCS and PN_BWpCS) cold stabilization, after second fermentation (T2M) and the final wines aged for nine months (T9M) samples.

Table 3.3: 2014 Robertson and Darling colorimetric results.

	Blends				T2M				T9M			
Robertson	**0ºC**	**10ºC**	**25ºC**	**30ºC**	**0ºC**	**10ºC**	**25ºC**	**30ºC**	**0ºC**	**10ºC**	**25ºC**	**30ºC**
TP	76.59e	81.28de	104.78ab	106.56a	88.11de	90.67cde	95.55abcd	104.28ab	88.11cde	85.61de	106.24ab	101.80abc
CI	0.100de	0.107de	0.170b	0.153bc	0.085e	0.094de	0.120cde	0.130cd	0.098de	0.109de	0.207a	0.178ab
CH	2.16de	1.82ef	1.38g	1.55fg	2.71ab	2.37bcd	2.32cd	2.14de	2.95a	2.71ab	2.41bcd	2.54bc
TH	0.223e	0.536de	1.353ab	1.398ab	0.182e	0.762cd	1.262abc	1.233abc	0.784cd	0.913bcd	1.647a	1.389ab
Darling	**0ºC**	**10ºC**	**25ºC**	**30ºC**	**0ºC**	**10ºC**	**25ºC**	**30ºC**	**0ºC**	**10ºC**	**25ºC**	**30ºC**
TP	76.18f	86.98ef	114.17abc	109.03bc	83.94ef	94.19de	111.38bc	103.80cd	75.57f	90.87e	123.58a	118.94ab
CI	0.107ef	0.164c	0.243b	0.232b	0.085f	0.127cdef	0.163cd	0.141cde	0.110def	0.173c	0.458a	0.275b
CH	1.85def	1.45ef	1.50def	1.25f	2.37bcde	1.99cdef	3.26ab	3.71a	2.83abc	2.32bcde	2.44bcd	2.05cdef
TH	0.085e	0.647cd	1.379ab	1.239b	0.156e	0.700cd	1.269b	0.738cd	0.467de	1.052bc	1.477ab	1.760a

Note: (Total phenolics in mg/L GAE, total hydroxycinnamates, colour intensity and colour hue) of Chardonnay/ Pinot noir blends, wines bottle aged for 2 months and 9 months (T2M and T9M).

3.3.3 Colorimetric analysis of 2015 vintage

The major cause of variation in the colorimetric data of Robertson (Figure 3.8) was due to the 25ºC treatments, which were problematic as previously indicated in 3.3.1. Colorimetric measurements of the 25ºC treatments were significantly higher than the other treatments throughout winemaking, accounting for about 5% of the variation in the data (Figure 3.8). In order to obtain a clear picture of the effect of the treatments on the colorimetric properties of Robertson wines, the 25ºC treatments were removed from the statistical analysis and the PCA biplot in Figure 3.9 produced. Removing the 25ºC outlying treatments revealed the same patterns observed in the 2014 data although the 2015 vintage had higher phenolics (Table 3.4). The variation between the remaining three treatments in the 2015 colorimetric data of Robertson (Figure 3.9, 54%) and Darling (Figure 3.10, 66%) was yet again due to the treatments. The 30ºC treatments again had higher TP, CI and TH than the lower temperature treatments (0ºC and 10ºC). With the exclusion of the 25ºC treatments the data showed a gradual increase in TP, CI, TH and a decrease in CH with greater temperature (Figures 3.9 and 3.10). The average total phenol content was lower than the range (176 – 195 mg/L GAE) found in literature (Chamkha *et al.*, 2003). The Chardonnay base wines again had significantly higher CH levels than Pinot noir base wines due to a lesser concentration of anthocyanins. Unlike the 2014 data, the Chardonnay base wines of 2015 were affected by the treatments. They had the same patterns in phenolics as the samples after blending. There were no significant differences in phenolics throughout winemaking for both vintages, similar to what has been found in literature on Cava (Gil-Muñoz *et al.*, 1999) but contradictory to other studies on Champagne (Stefenon *et al.,* 2013) and alternative varieties (Martínez-Lapuente *et al.,* 2013) which found a decrease in phenolics after the second fermentation.

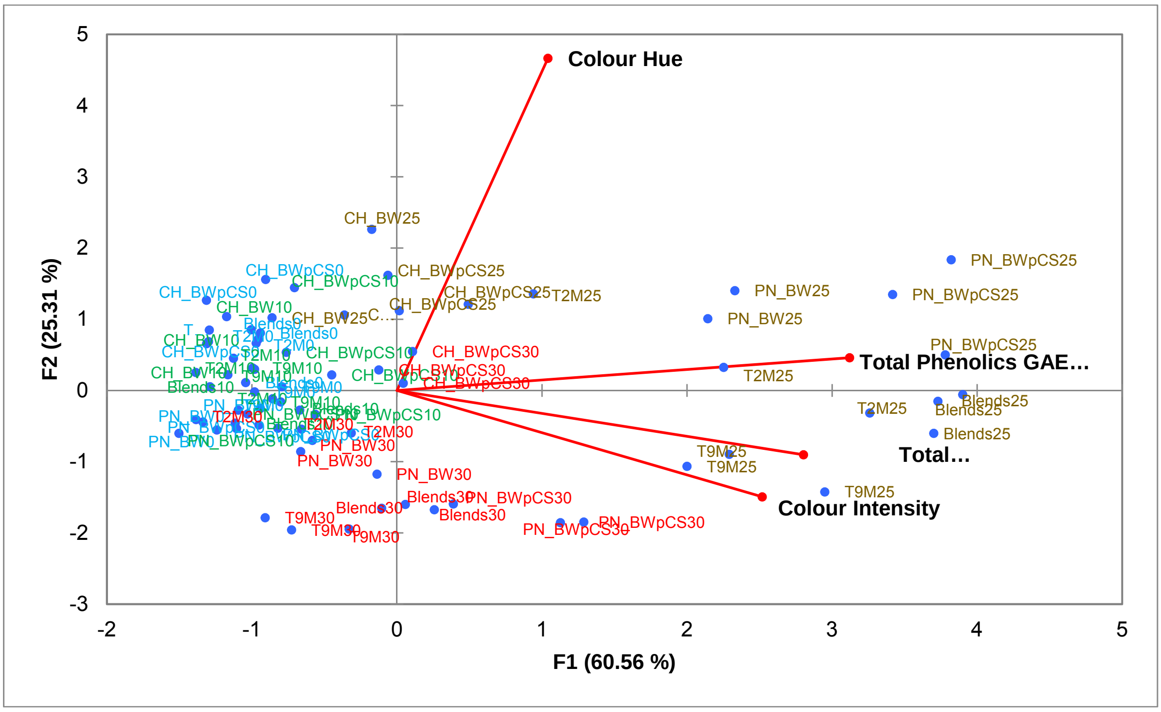

Figure 3.8. PCA biplot of Robertson 2015 colorimetric analysis (Colour hue, colour intensity, total phenolics in mg/l GAE, total hydroxycinnamates) results. Wines sampled before (CH_BW and PN_BW) and after (CH_BWpCS and PN_BWpCS) cold stabilization, after second fermentation (T2M) and the final wines aged for nine months (T9M) samples

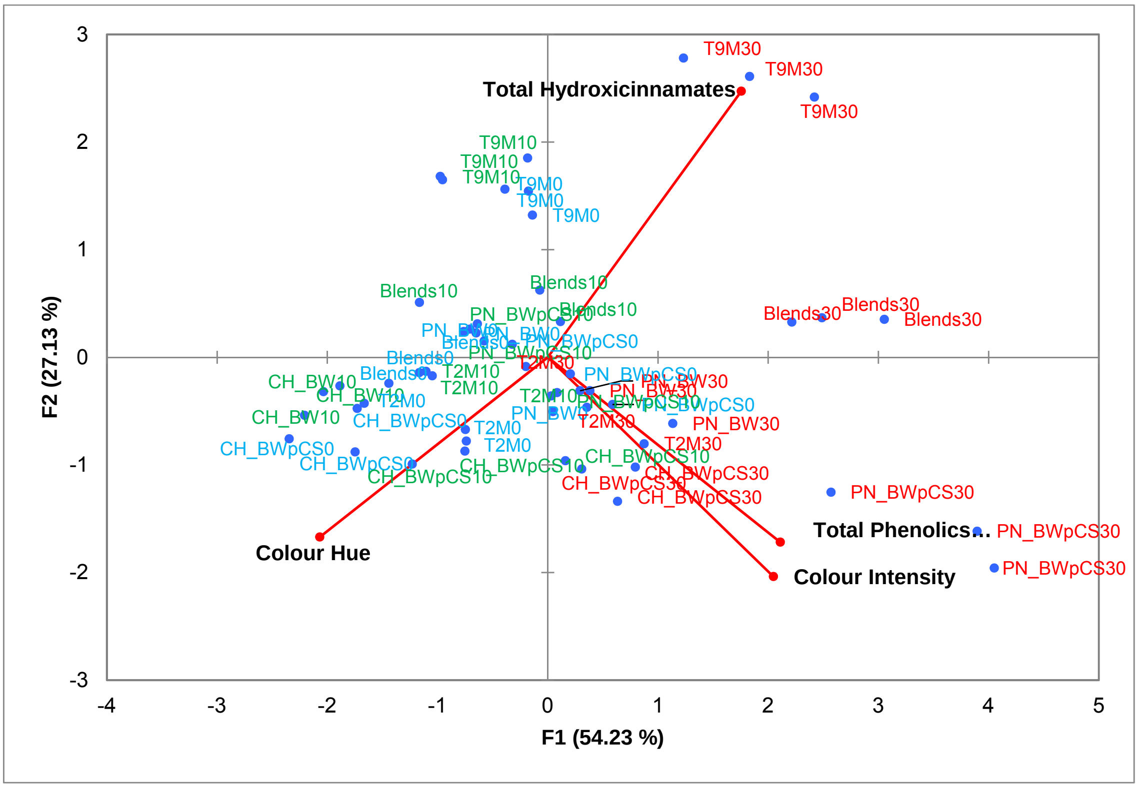

Figure 3.9. PCA biplot of Robertson 2015 colorimetric analysis (Colour hue, colour intensity, total phenolics in mg/L GAE, total hydroxycinnamates) results excluding the outlying 25ºC treatments. Wines sampled before (CH_BW and PN_BW) and after (CH_BWpCS and PN_BWpCS) cold stabilization, after second fermentation (T2M) and the final wines aged for nine months (T9M) samples

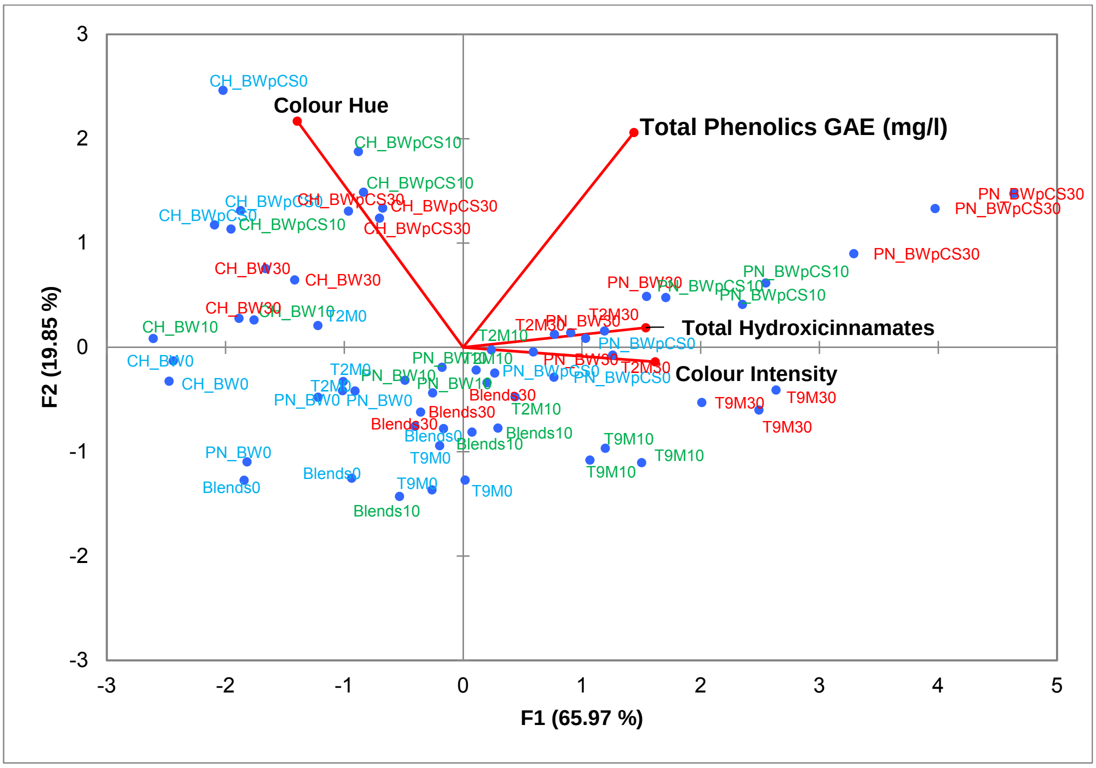

Figure 3.10. PCA biplot of Darling 2015 colorimetric analysis (Colour hue, colour intensity, total phenolics in mg/l GAE, total hydroxycinnamates) results. Wines sampled before (CH_BW and PN_BW) and after (CH_BWpCS and PN_BWpCS) cold stabilization, after second fermentation (T2M) and the final wines aged for nine months (T9M) samples.

Table 3.4: 2015 Robertson and Darling colorimetric results.

	Blends			T2M			T9M		
Robertson	**0ºC**	**10ºC**	**30ºC**	**0ºC**	**10ºC**	**30ºC**	**0ºC**	**10ºC**	**30ºC**
TP	110.16bcd	107.33cd	143.16a	123.54b	115.56bc	123.01b	108.89bcd	96.09d	115.63bc
CI	0.191cde	0.230c	0.411a	0.169de	0.213cd	0.33b	0.134e	0.134e	0.211cd
CH	3.00a	2.54cd	1.90e	3.03a	2.75bc	2.42d	2.60bcd	2.76b	1.58f
TH	0.992cd	1.250c	2.218b	0.602e	0.767de	0.871de	2.124b	2.245b	2.94a
Darling	**0ºC**	**10ºC**	**30ºC**	**0ºC**	**10ºC**	**30ºC**	**0ºC**	**10ºC**	**30ºC**
TP	120.06e	143.45cde	145.30cd	154.86c	182.26ab	204.04a	128.61de	163.50bc	199.04a
CI	0.389cd	0.524b	0.630a	0.357d	0.520b	0.646a	0.282e	0.440c	0.602a
CH	2.45b	2.21c	2.74a	2.95a	2.45b	2.46b	2.06c	1.65d	1.70d
TH	1.292d	1.529cd	1.206d	1.177d	1.672cd	2.033c	2.793b	3.389a	3.903a

Note: (Total phenolics in mg/l GAE, total hydroxycinnamates, colour intensity and colour hue) of Chardonnay/ Pinot noir blends, wines bottle aged for 2 months and 9 months (T2M and T9M).

3.4 Conclusion

The storage of grapes at different temperatures had an effect on the extraction of phenolics. Grapes stored at lower temperature (0 and 10ºC) had lower phenolic content than grapes stored at higher temperatures (25 and 30ºC). The high storage temperatures (25 and 30ºC) allowed for better extraction of the phenolics into the free-run. This may have been due to greater enzyme activity at higher temperatures, which in turn leads to cell breaking and subsequent extraction of phenolics into the juice. The total phenolics (GAE), colour intensity and total hydroxycinnamates were all higher in wines made from grapes stored at higher temperatures. Hence, there is better extraction of phenolics at higher temperature than at lower temperatures, which is not desired by TSW winemakers. Similar to a study on Champagne, the phenolic content did not change throughout winemaking showing the stability of the phenolics during TSW winemaking.

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Chapter 4

The effect of grape storage temperature on the sensory attributes of Méthode Cap Classique wines

4.1 Introduction

Sensory evaluation of wine is very important because it ultimately connects viticulture and winemaking to the consumer (Kerslake *et al.*, 2013). Although chemical analysis is also very important, it cannot always be connected to the sensory perception of wines due to the complicated matrix effect of wine. The sensory experience of traditional sparkling wine (TSW) is very different from that of still wine. This is primarily due to the effervescent nature of TSW which although is desirable, can be problematic when performing standardised sensory evaluations. It is thus important to preserve the effervescence as it can affect both the aroma and taste of TSWs. The effervesce provides the desired mouthfeel attributes such as fresh or crisp acidity (Vannier, Brun & Feinberg, 1999). The effervescence also contributes to the aroma perception of TSW. The aroma of TSW is comprised of grape, fermentation and aging-derived attributes. The closed fermentation in the bottle and aging on the less provides most of the aroma attributes such as creamy, oak, yeasty and autolytic character, classically associated with TSW (Ganss *et al.*, 2011). There is no published or recommended method for the sensory evaluation of TSW but guidelines have fairly recently been provided for how to ensure uniformity in the evaluation of TSW across judges when taking into account the effervescence (Buxaderas *et al.*, 2010; Hood-White *et al.*, 2015). This study used sorting analysis to try to distinguish between the different treatments and this has previously been successively used in beers (Chollet *et al.*, 2011; Chollet *et al.*, 2014).

For this study, we hypothesize that the grape storage temperature will affect the aroma and the mouthfeel of the final (T9M) wines. This could possibly be due to different levels of extraction of the aroma compound precursors and other compounds that can affect the taste and mouthfeel, such as phenolics. An investigation into the link between chemical composition and sensory is beyond the scope of this study.

4.2 Materials and methods

4.2.1 Vinification and Sampling

See 3.2.1

4.2.2 Sensory evaluation

A free sorting exercise was performed on MCCs (aged for 9-months in the bottle - T9M) made from grapes harvested from two farms (Robertson and Darling). The grapes were stored at 0, 10, 25 and 30ºC and processed in triplicate. Using a panel of 30 expert judges, 12 wines were assessed in 2014 and 9 wines in 2015. Free sorting was chosen as the method of evaluation because it has previously been used on beer which has effervescence similar to MCCs (Chollet & Valentin, 2011). The tasting was performed in two sessions (a morning and an afternoon session each with 15 judges) over two days (one day for Robertson and another for Darling). The samples were coded with a unique 3-digit number and randomised according to each judge. Different codes were presented for the aroma flight and the taste flight. Approximately 20 ml of each wine was poured into black-tinted tasting glasses and covered with a petri dish. The aroma flight was presented first and the taste flight second with a 15-minute intermission between flights. The judges were instructed to smell/ taste the wines (Figure B1), group them according to their similarities and make a list of the similar attributes of each group with a choice to provide individual descriptors for each wine if compelled to do so. The free sorting exercise performed in 2014 generated aroma and taste descriptors which were used for the 2015 sorting exercise (Figure B2).

4.2.3 Statistical analysis

Co-occurrence matrices were generated for the groupings of the wines and for the attributes for each judges. Contingency matrices were used to calculate statistical relationships between treatments and aroma/ taste attributes using XLSTAT. Correspondence analysis (CA) was used to visualize the relationship between the treatments and the aroma/ taste attributes. Agglomerative Hierarchical Clustering (AHC) was used to find significant grouping of treatments.

4.3 Results and discussions

Since the objective of the study was to evaluate the effect of grape storage temperature on the sensory profile of MCCs, all other causes of variation, such as grape origin, were excluded. Therefore, the wines from the two farms were evaluated separately. The results from the aroma sorting are presented first followed by the taste sorting results, according to vintage. Statistical results are presented in the following order: scatter plots representing the samples and associated attributes, dendrograms showing the grouping of the samples, and frequency of citation tables for the groups of samples (according to the dendrograms).

4.3.1 Sensory evaluation of 2014 Méthode Cap Classique wines

The Agglomerative Hierarchical Clustering (AHC) dendrograms of the Darling and Robertson MCCs (Figure 4.2 and 4.4, respectively) aroma sorting showed clear groupings according to temperature. Multidimensional scaling (MDS) showed close grouping of the wines according to temperature with a Kruskal's test index of 0.095 for Darling and 0.161 for Robertson. This meant that judges observed clearer similarities between temperature groups in the aroma profile of Darling wines than they did for Robertson wines. There was good repeatability between biological repeats with the exception of one of the 30ºC repeats in each farm. The treatments accounted for 59% of the observed variance in the Darling aroma sorting CA plots (Figure 4.1) and only 33% for Robertson (Figure 4.3). Lower temperature treatments had more positive aroma attributes compared to higher temperature treatments (Tables 4.1 and 4.2). They were more frequently cited as being fruitier, fresher, generally more aromatically appealing and in line with attributes cited for other young (9-months old) traditional sparkling wines (Vannier *et al.*, 1999; Torrens *et al.*, 2010). Higher temperature treatments had attributes such as toasty, oaky, buttery and vanilla which were positively similar to those previously cited for 18-months and older traditional sparkling wines (De la Presa-Owens *et al.*, 1998). The higher temperature treatments also had negative aromas such as oxidation, chemical and solvent-like aromas which have previously been linked to oxidised white wines (Silva Ferreira *et al.*, 2003). Negative attributes such as sulphur-related, VA, solvent-like and oxidation were the most frequently cited in correlation to the higher temperature treatments. The outlying 30_R3 treatment in Darling which was described as vegetative, an attribute that has previously been associated with the oxidation of white wines implying that this particular treatment was exceptionally oxidised (Silva Ferreira *et al.*, 2003).

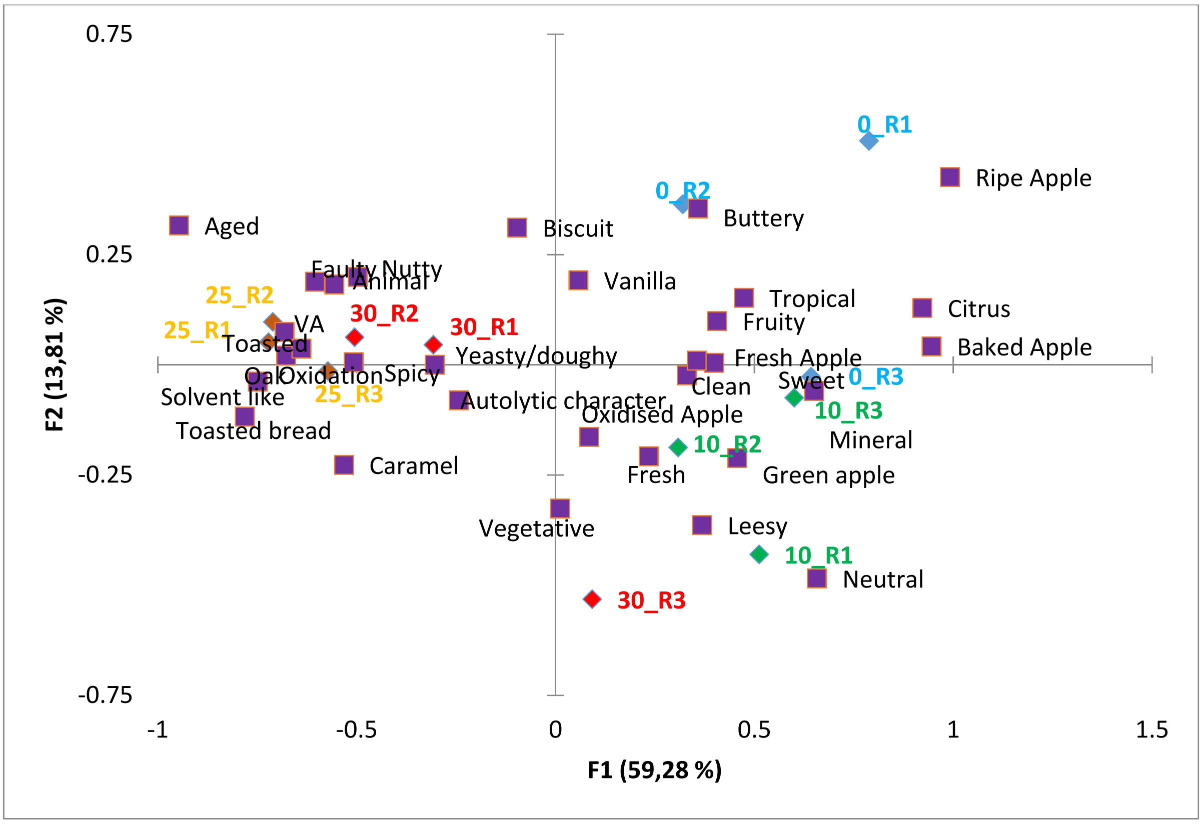

Figure 4.10. Correspondence analysis (CA) conducted on aroma sorting results of MCCs made from grapes harvested from Darling in 2014 and stored at 0, 10, 25 and 30ºC with three repeats per temperature

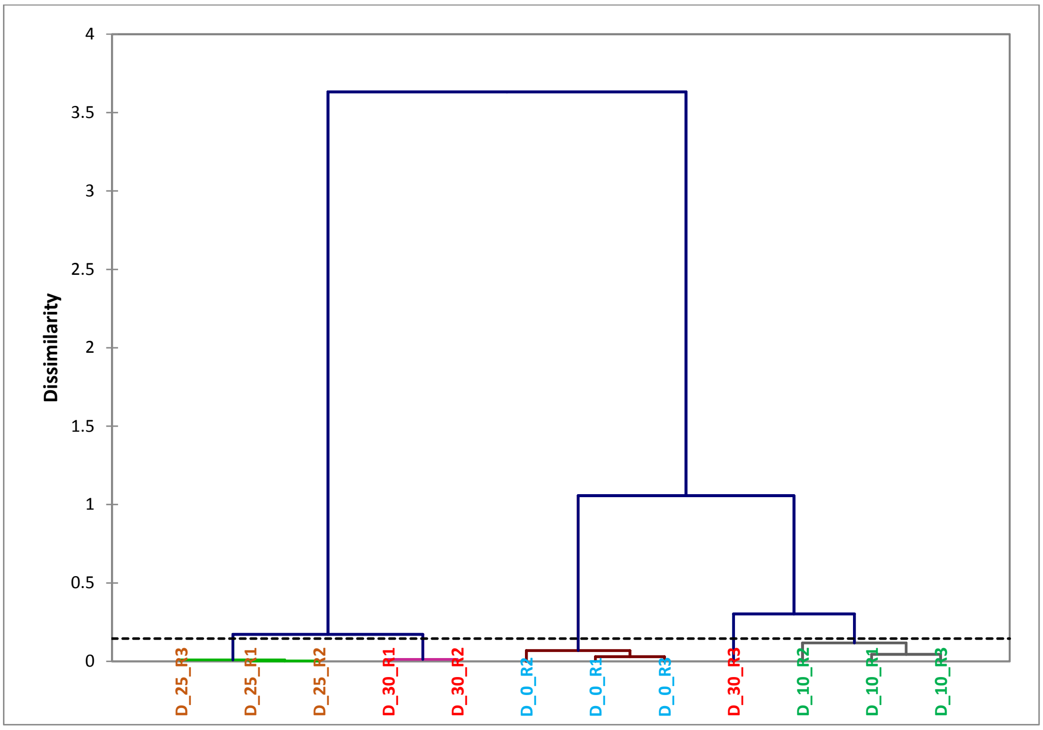

Figure 4.2. Dendrogram of the Agglomerative Hierarchical Clustering (AHC) conducted on 2014 Darling aroma sorting of MCCs made from grapes stored at 0, 10, 25 and 30ºC with three biological repeats per treatment

Table 4.1: 2014 Darling frequency table of aroma attributes ranked from highest to lowest. The samples are grouped according to the AHC (Figure 4.2).

	Solvent like	VA	Faulty	Oxidation	Yeasty/doughy	Toasted Oak
D_25_R1	9	7	6	5	4	3
D_25_R2	9	7	6	4	5	3
D_25_R3	7	6	6	6	3	3
Sum	**25**	**20**	**18**	**15**	**12**	**9**
	Solvent like	**Faulty**	**VA**	**Fruity**	**Oxidation**	**Vegetative**
D_30_R1	4	6	7	5	4	3
D_30_R2	8	5	3	4	3	3
Sum	**12**	**11**	**10**	**9**	**7**	**6**
	Fruity	**Fresh Apple**	**Ripe Apple**	**Citrus**	**Red fruits**	**Fresh**
D_0_R1	9	5	5	5	5	3
D_0_R2	5	3	3	3	4	2
D_0_R3	7	5	5	3	1	5
Sum	**21**	**13**	**13**	**11**	**10**	**10**
	Fruity	**VA**	**Citrus**	**Green apple**	**Neutral**	**Fresh**
D_10_R1	6	2	4	5	5	4
D_10_R2	2	1	4	3	3	3
D_10_R3	5	9	4	4	3	3
Sum	**13**	**12**	**12**	**12**	**11**	**10**
	Fresh Apple	**Fruity**	**Fresh**	**Vegetative**	**Solvent like**	**Yeasty/doughy**
D_30_R3	4	4	4	4	3	3

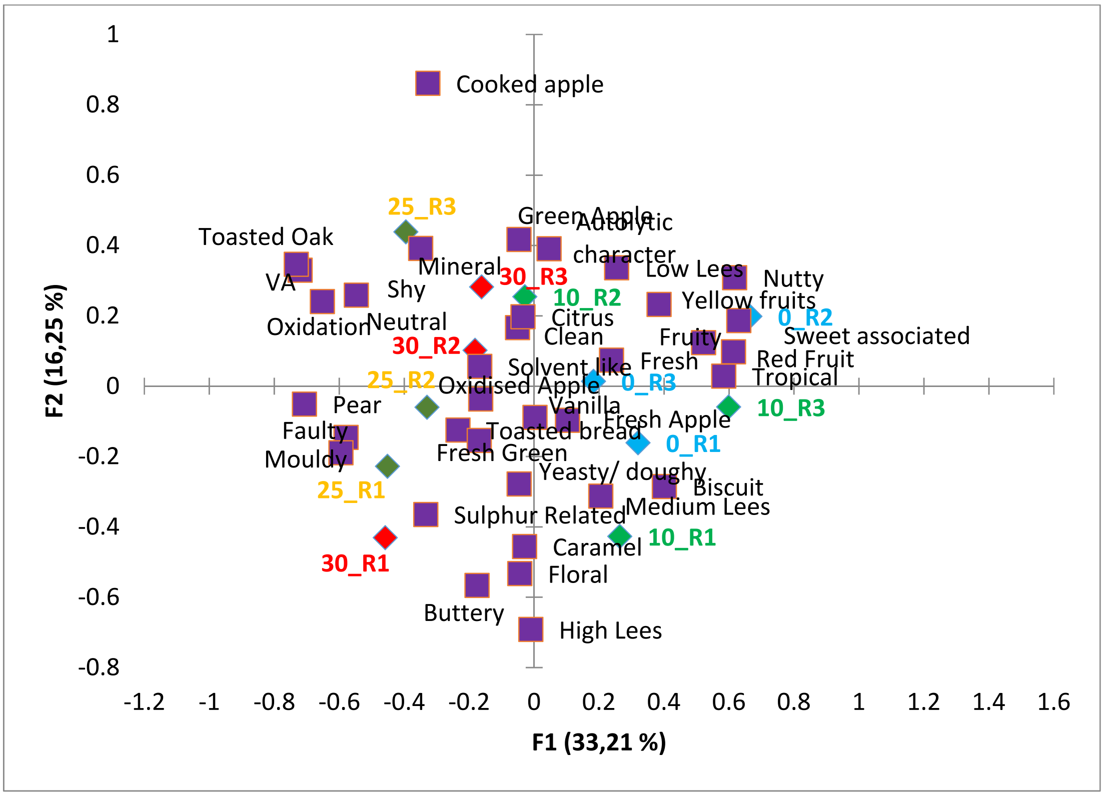

Figure 4.3: Correspondence analysis (CA) conducted on aroma sorting results of MCCs made from grapes harvested from Robertson in 2014 and stored at 0, 10, 25 and 30ºC with three repeats per temperature.

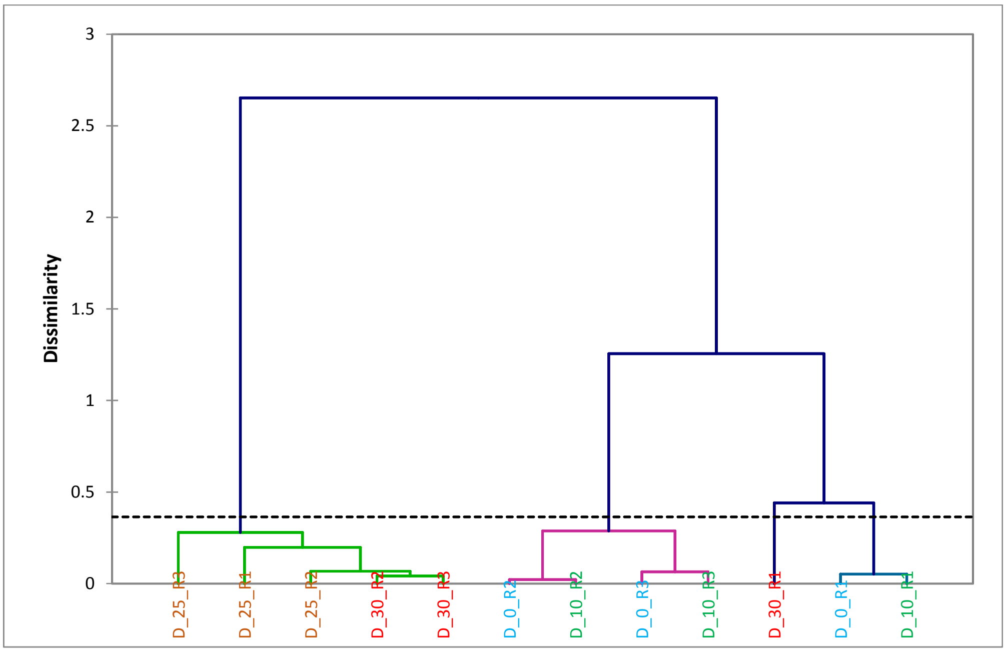

Figure 4.4: Dendrogram of the Agglomerative Hierarchical Clustering (AHC) conducted on 2014 Robertson aroma sorting of MCCs made from grapes stored at 0, 10, 25 and 30ºC with three biological repeats per treatment.

Table 4.2: 2014 Robertson frequency table of aroma attributes ranked from highest to lowest. The samples are grouped according to the AHC (Figure 4.4).

	Yeasty/ doughy	Fresh Apple	Fresh	Toasted bread	VA	Sulphur Related
R_25_R1	7	5	0	4	2	4
R_25_R2	8	7	3	3	4	1
R_25_R3	3	4	4	2	3	1
R_30_R2	5	6	8	2	1	4
R_30_R3	6	4	7	5	3	3
Sum	**29**	**26**	**22**	**16**	**13**	**13**
	Fresh Apple	**Fresh**	**Yeasty/ doughy**	**Fruity**	**Sweet associated**	**Tropical**
R_0_R1	5	7	7	4	5	2
R_0_R2	6	7	4	7	6	5
R_0_R3	4	4	6	5	3	2
R_10_R1	7	4	8	3	2	3
R_10_R2	6	3	1	4	4	1
R_10_R3	8	7	4	6	4	7
Sum	**36**	**32**	**30**	**29**	**24**	**20**

The Agglomerative Hierarchical Clustering (AHC) dendrogram of Darling (Figure 4.6) and Robertson (Figure 4.8) taste sorting showed grouping of the MCCs according to temperature treatments, similar to the aroma sorting results. MDS showed that the judges were not able to group the MCCs according to taste as well as they did in the aroma sorting. Similar to the aroma sorting, Kruskal's test index for Darling (0.194) showed better grouping of the MCCs according to temperature treatments compared to Robertson (0.215). Due to retronasal perceptions by the judges, most of the attributes were similar to those generated for the Darling aroma sorting. The judges were, however, consistent in generating similar results for the taste as for the aroma in correlation to the groups of treatments (Figure 4.5). In terms of mouthfeel and flavour judges cited Darling lower temperature treatments as being more balanced, well developed, full bodied with higher acidity, and higher temperature treatments were found to be acidic/sour, more bitter, sweeter with a longer after-taste (Table 4.3). The Robertson data (Figure 4.7) shows that upon tasting judges were unable to distinguish the wines according to treatments even given retronasal aroma perceptions. The attributes generated for the wines were more positive than those cited for the Darling wines (Table 4.4).

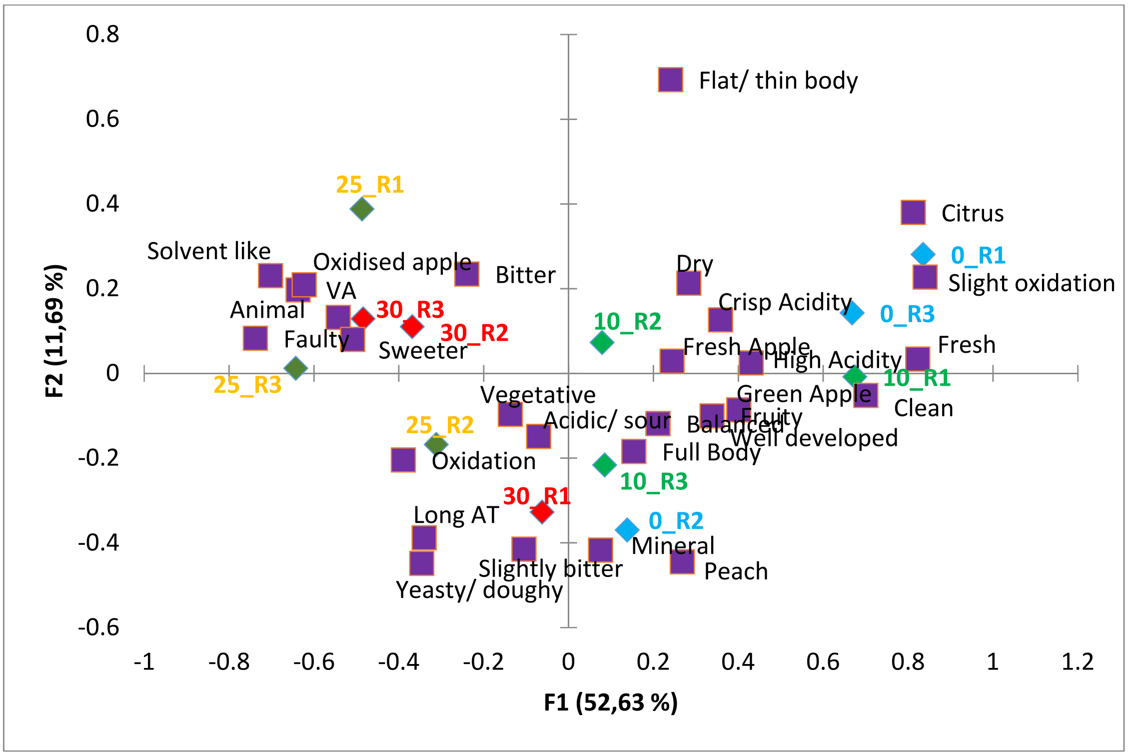

Figure 4.5: Correspondence analysis (CA) conducted on taste sorting results of MCCs made from grapes harvested from Darling in 2014 and stored at 0, 10, 25 and 30ºC with three repeats per temperature.

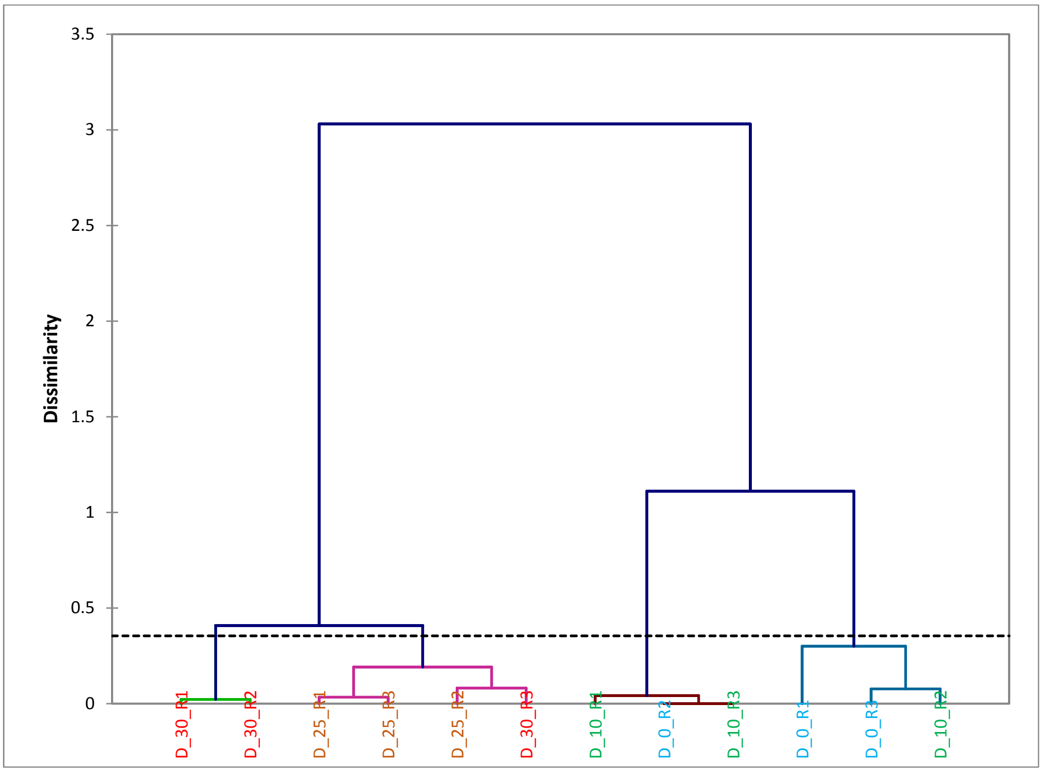

Figure 4.6: Dendrogram of the Agglomerative Hierarchical Clustering (AHC) conducted on 2014 Darling taste sorting of MCCs made from grapes stored at 0, 10, 25 and 30ºC with three biological repeats per treatment.

Table 4.3: 2014 Darling frequency table of taste attributes ranked from highest to lowest. The samples are grouped according to the AHC (Figure 4.6).

	VA	**Faulty**	**Acidic/ sour**	**Solvent like**	**Yeasty/ doughy**	**Bitter**
D_25_R1	9	4	2	5	1	4
D_25_R2	8	2	3	3	4	2
D_25_R3	7	5	3	5	3	2
D_30_R1	4	4	6	0	6	2
D_30_R2	6	6	5	3	3	3
D_30_R3	9	3	4	4	2	4
Sum	**43**	**24**	**23**	**20**	**19**	**17**
	High Acidity	**Fresh Apple**	**Fruity**	**Green Apple**	**Acidic/ sour**	**Citrus**
D_0_R1	5	5	3	4	2	5
D_0_R2	4	3	3	3	4	1
D_0_R3	5	4	4	3	3	4
D_10_R1	5	5	4	4	3	3
D_10_R2	3	4	4	3	3	2
D_10_R3	5	4	2	3	2	1
Sum	**27**	**25**	**20**	**20**	**17**	**16**

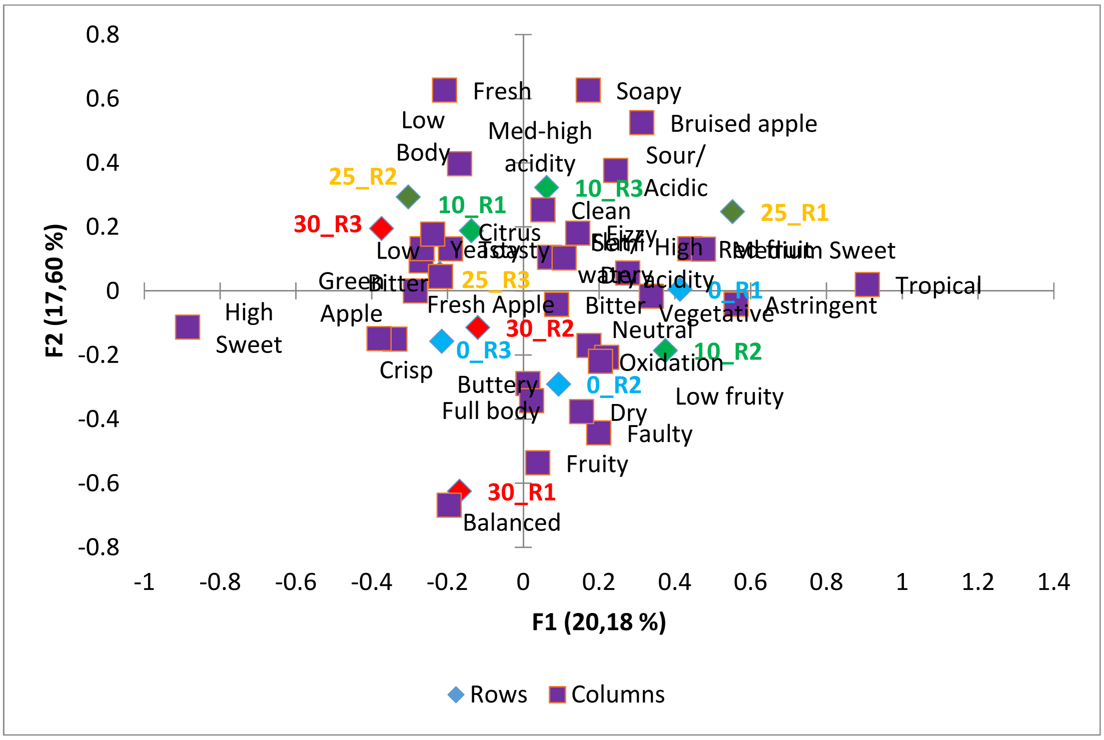

Figure 4.7: Scatter plot of taste sorting results of MCCs made from grapes harvested from Robertson in 2014 and stored at 0, 10 and 30ºC with three repeats per temperature.

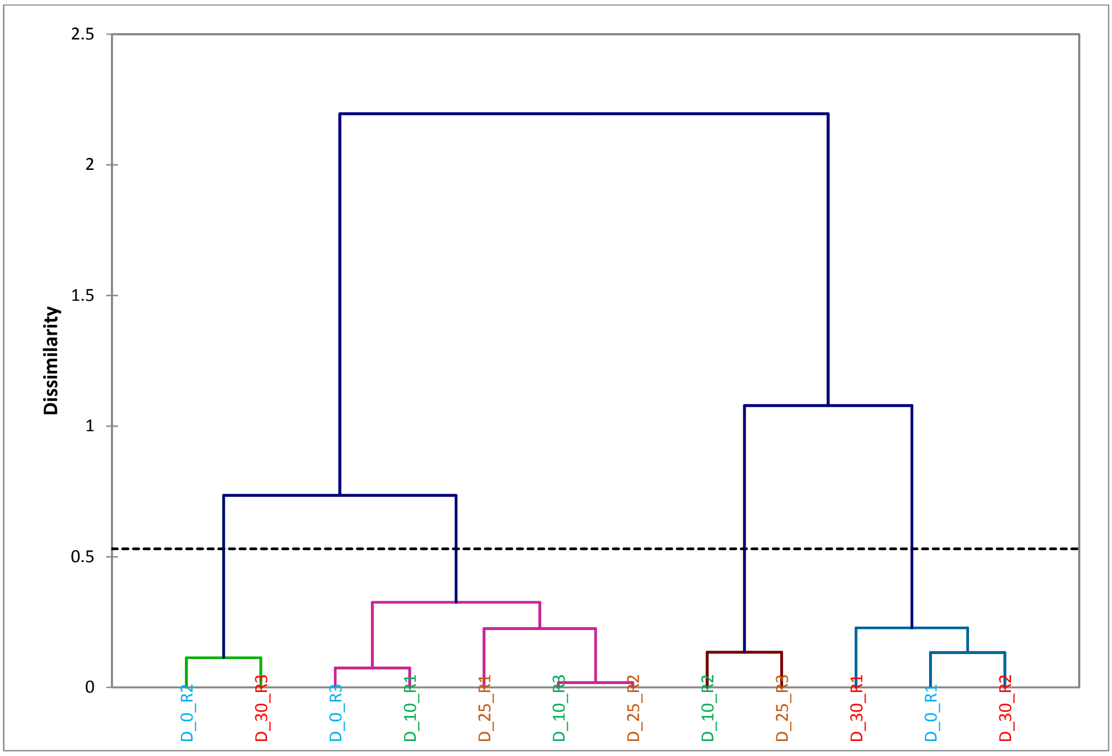

Figure 4.8: Dendrogram of the Agglomerative Hierarchical Clustering (AHC) conducted on 2014 Robertson taste sorting of MCCs made from grapes stored at 0, 10, 25 and 30ºC with three biological repeats per treatment.

Table 4.4: 2014 Robertson frequency table of taste attributes ranked from highest to lowest.

	Yeasty	Bitter	Crisp	Fresh Apple	Sour/Acidic	Fruity
R_0_R1	2	5	2	2	3	3
R_0_R2	5	2	2	5	2	5
R_0_R3	4	5	3	2	1	2
R_10_R1	5	4	2	2	3	1
R_10_R2	3	4	2	0	3	2
R_10_R3	4	3	2	4	5	2
R_25_R1	3	3	0	1	4	1
R_25_R2	7	4	4	4	3	0
R_25_R3	5	4	5	2	2	2
R_30_R1	3	3	4	3	0	5
R_30_R2	3	3	4	3	2	2
R_30_R3	7	0	3	4	2	2
Sum	**51**	**40**	**33**	**32**	**30**	**27**

4.3.2 Sensory evaluation of 2015 Méthode Cap Classique wines

Due to vinification difficulties discussed in Chapter 3.3.1, the 25ºC treatments were removed from sensory evaluation after a preliminary screening revealed the wines to be very oxidised hence the 2015 sorting exercises were performed on 9 wines. The Agglomerative Hierarchical Clustering (AHC) dendrograms of the Darling and Robertson MCCs (Figure 4.10 and 4.12, respectively) aroma sorting showed clear groupings according to temperature. Multidimensional scaling (MDS) showed close grouping of the wines according to temperature with a Kruskal's test index of 0.123 for Darling and 0.108 for Robertson. These indices are larger than those calculated in 2014 meaning that judges were less able to group the 2015 MCCs according to temperature treatments. The sorting analysis for 2014 wines was on 12 wines at a time and 9 were assessed in 2015. The differences in the number of wines assessed at a time has been shown to influence the statistical coherence of results with 12 being the optimum number for achieving results that are more statistically confident (Chollet *et al.*, 2011; Chollet *et al.*, 2014).

The lower temperature treatments (0 and 10ºC) were again associated with positive aroma attributes but these positive aroma attributes were more frequently cited than negative attributes for all treatments compared to 2014 where negative attributes more frequently cited (Tables 4.5 and 4.6). The Darling 0ºC treatments were described by judges as having green and baked apple aromas, similar to the 2014 vintage, whilst the Robertson 0ºC treatments were associated with oxidation and toasted bread notes (aside from the 0_R3 outlier), showing very different attributes for the treatments between the two farms. The 10ºC treatments of Darling (Figure 4.9) were perceived by judges to be more fresh and fruity, similar to the 2014 aroma sorting results (Figures 4.1 and 4.3). The Robertson (Figure 4.11) lower temperature treatments, however, were perceived as having attributes similar to those previously associated with higher temperature treatments (ex. VA, vanilla, caramel). The Darling 30ºC treatments again grouped with negative attributes such as solvent-like, sulphur-related, VA and faulty (Figure 4.9 and 4.10) whilst the Robertson 30ºC treatments were associated with fruity notes such as green apple, yellow fruit, pear and freshness (Figure 4.11 and 4.12), notes associated with lower temperature treatments in the previous vintage and for 2015 Darling aroma profile.

Not similar to the 2014 vintage is that the wines made from lower temperature treatments were associated with toasty and caramel notes which were previously associated with the higher temperature treatments and commonly associated with older (18-months or more) Cap Classique wines (De la Presa-Owens *et al.*, 1998). Vintage differences are very evident in the sensory profile of MCCs and although the vintages are not the same, there are still significant differences between the temperature treatments. In this work, the final MCC was not blended in order to achieve sensorial uniformity, which is a common commercial practice for Champagne. Robertson 2015 aroma profile was very unique in that mature Cap Classique attributes such as creamy, vanilla, yeasty and buttery were cited throughout all the treatments with 30ºC treatments being more associable with fruity/ fresh aromas.

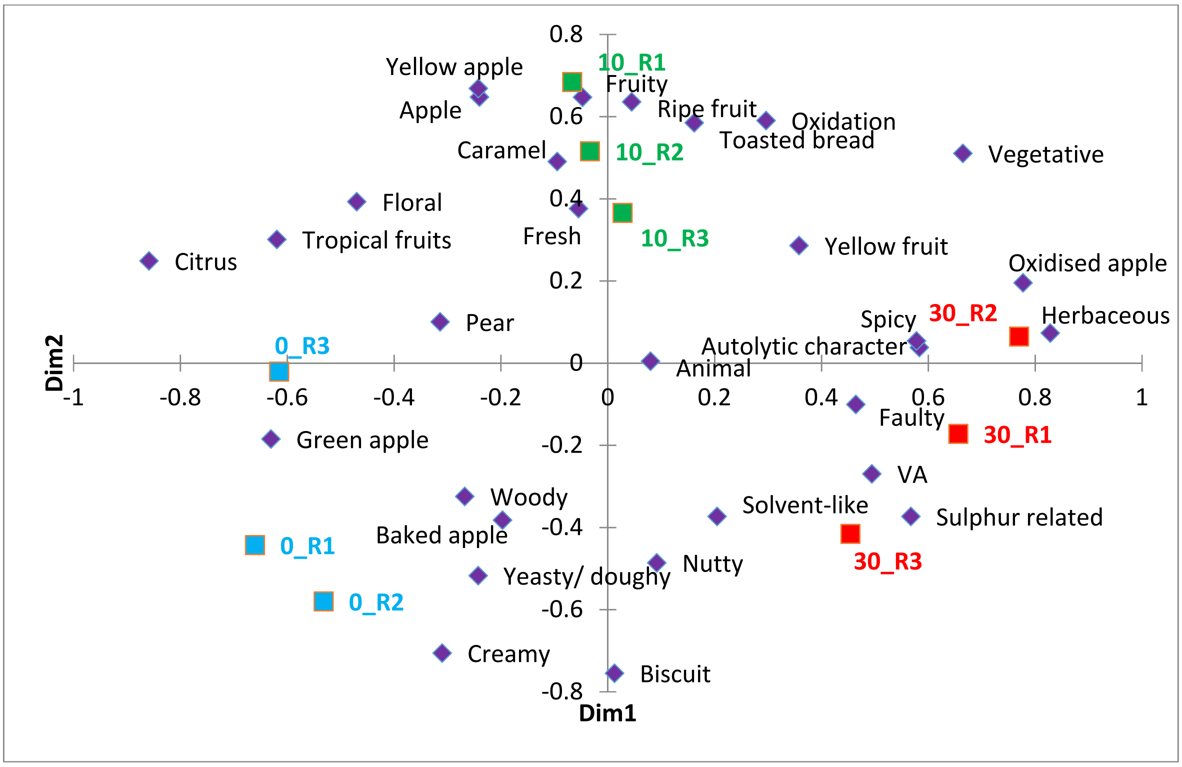

Figure 4.9: Scatter plot of aroma sorting results of MCCs made from grapes harvested from Darling in 2015 and stored at 0, 10 and 30ºC with three repeats per temperature.

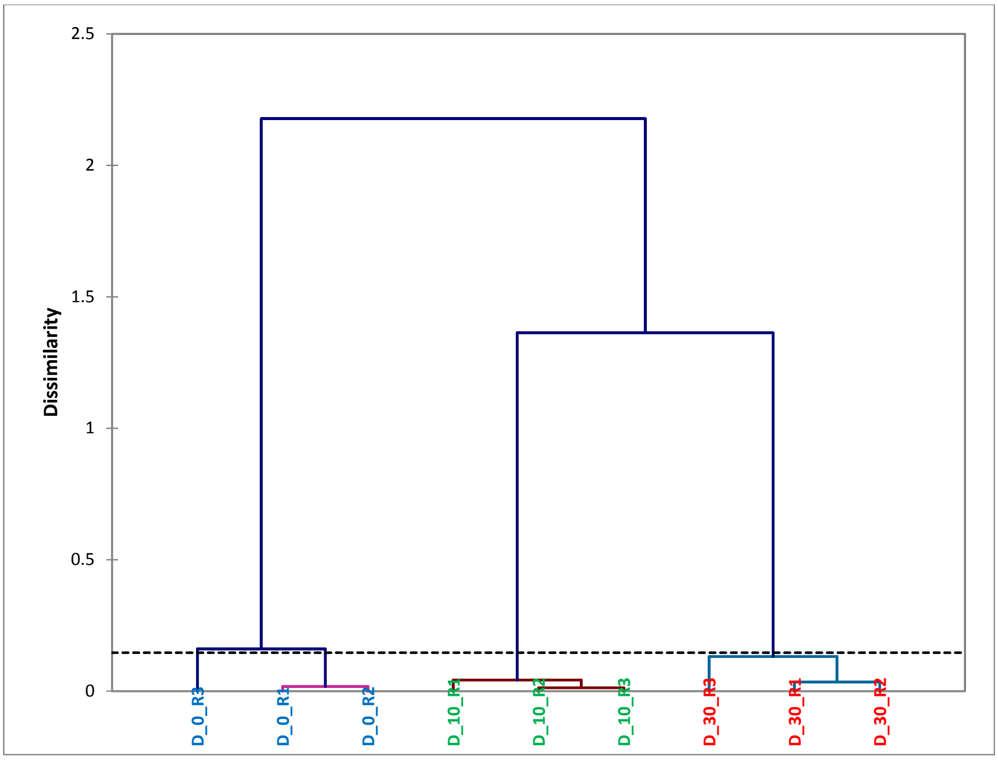

Figure 4.10: Dendrogram of the Agglomerative Hierarchical Clustering (AHC) conducted on 2015 Darling aroma sorting of MCCs made from grapes stored at 0, 10 and 30ºC with three biological repeats per treatment.

Table 4.5: 2015 Darling frequency table of aroma attributes ranked from highest to lowest. The samples are grouped according to the AHC (Figure 4.10).

	Yeasty/ doughy	Tropical fruits	Citrus	Fruity	Toasted bread	Nutty
D_0_R1	4	4	3	1	2	3
D_0_R2	3	3	3	3	2	2
D_0_R3	6	3	3	3	3	1
Sum	**13**	**10**	**9**	**7**	**7**	**6**
	Tropical fruits	**Fruity**	**Toasted bread**	**Vegetative**	**Caramel**	**Citrus**
D_10_R1	4	4	2	3	3	4
D_10_R2	3	5	6	2	5	2
D_10_R3	6	3	4	5	1	2
Sum	**13**	**12**	**12**	**10**	**9**	**8**
	Oxidised apple	**VA**	**Yeasty/ doughy**	**Vegetative**	**Solvent-like**	**Toasted bread**
D_30_R1	6	2	4	4	5	3
D_30_R2	5	7	3	4	0	4
D_30_R3	2	3	4	2	4	1
Sum	**13**	**12**	**11**	**10**	**9**	**8**

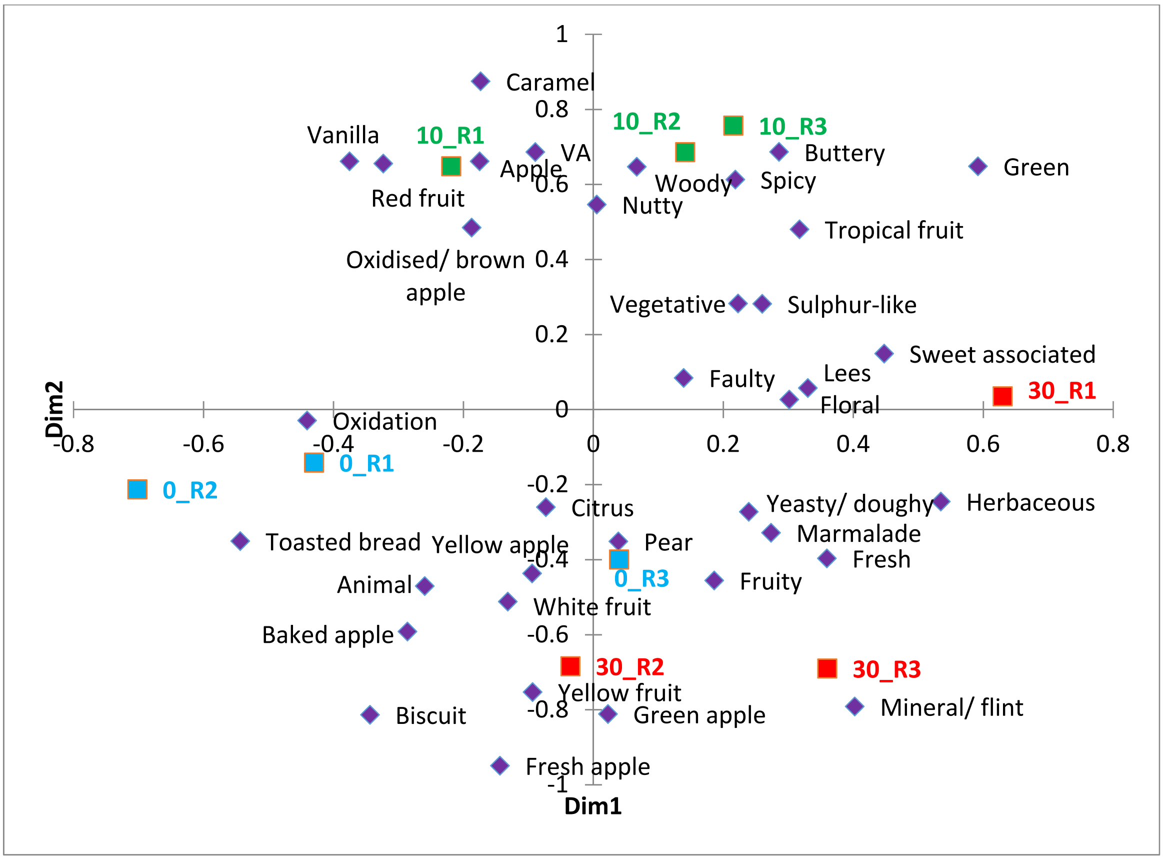

Figure 4.11: Scatter plot of aroma sorting results of MCCs made from grapes harvested from Robertson in 2015 and stored at 0, 10 and 30ºC with three repeats per temperature.

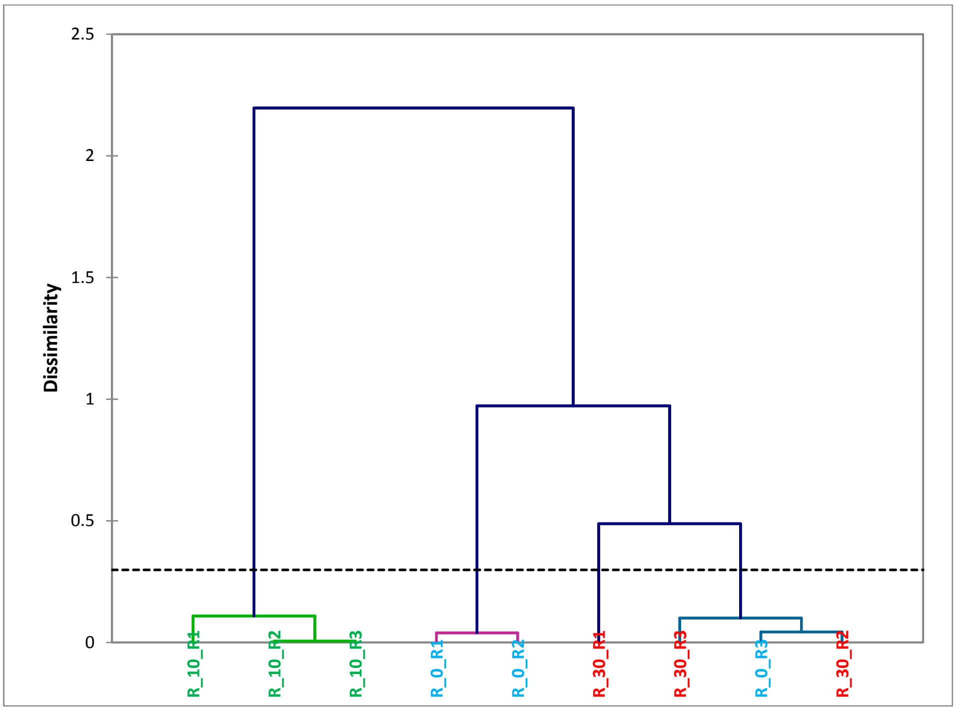

Figure 4.12: Dendrogram of the Agglomerative Hierarchical Clustering (AHC) conducted on 2015 Robertson aroma sorting of MCCs made from grapes stored at 0, 10 and 30ºC with three biological repeats per treatment.

Table 4.6: 2015 Robertson frequency table of aroma attributes ranked from highest to lowest. The samples are grouped according to the AHC (Figure 4.12).

	Yeasty/ doughy	**Oxidised/ brown apple**	**VA**	**Caramel**	**Citrus**	**Red fruit**
R_10_R1	5	4	3	2	2	4
R_10_R2	4	5	2	3	3	2
R_10_R3	4	2	5	4	3	2
Sum	**13**	**11**	**10**	**9**	**8**	**8**
	Yeasty/ doughy	**Biscuit**	**Fruity**	**Toasted bread**	**White fruit**	**ised/ brown a**
R_0_R1	3	4	2	4	4	3
R_0_R2	6	2	4	2	2	3
Sum	**9**	**6**	**6**	**6**	**6**	**6**
	Yeasty/ doughy	**Pear**	**Fruity**	**Citrus**	**Fresh**	**Green apple**
R_0_R3	4	5	5	5	5	3
R_30_R2	6	3	4	3	3	3
R_30_R3	5	4	2	3	2	3
Sum	**15**	**12**	**11**	**11**	**10**	**9**
	Yeasty/ doughy	**Fruity**	**Sulphur-like**	**Fresh**	**White fruit**	**Nutty**
R_30_R1	7	6	5	4	3	3

The sorting of Robertson (Figure 4.15) and Darling (Figure 4.13) MCCs according to taste again showed that judges were less able to distinguish between treatments according to taste, similar to 2014. AHC of Darling (Figure 4.14) and Robertson (Figure 4.16) grouping of the wines with poor repeatability between biological repeats, revealed in the MDS by high Kruskal's test index for both Darling (0.185) and Robertson (0.183). A scatter plot of 2015 Darling taste sorting results (Figure 4.13) showed that 0ºC treatments were similar to those of the aroma of both 0 and 10ºC treatments in its fresh and fruity attributes due to retronasal perception. Mouthfeel attributes such as clean, dry and medium were associated with 0 and 10ºC, similar to the 2014 taste sorting.

The scatter plot also showed that the 2015 taste sorting of the 30°C treatment is similar to the 2014 taste attributes (oxidation, bitterness and full body) but lacks in retronasal attribute (buttery, caramel and vanilla). This matches the aroma profile of the 30°C that only showed negative attributes and no matured Méthode Cap Classique attributes shown in 2014. Similar to 2014 results, one of the 30°C treatments (30_R2) correlated with vegetative attribute. The taste sorting exercise for Robertson 2015 (Figure 4.13 and 4.14) shows a scattering similar the 2014 vintage (Figure 4.7 and 4.8) but to a lesser extent. Similar to the 2015 results for both the aroma and taste components, less negative attributes were cited in comparison to the 2014 vintage (Tables 4.7 and 4.8). The study additionally showed that regardless of the judges' level of expertise in sparkling wine; they were able to detect differences in the wines according to the temperature treatments.

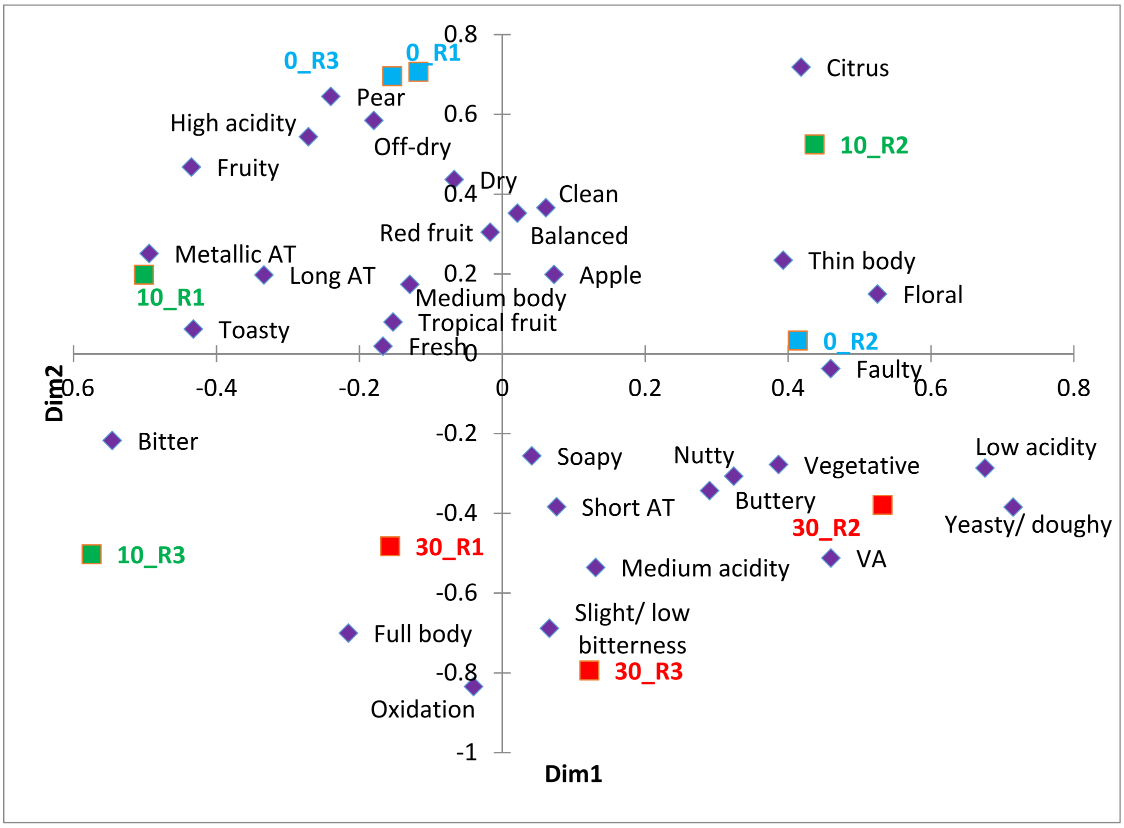

Figure 4.13: Scatter plot of taste sorting results of MCCs made from grapes harvested from Darling in 2015 and stored at 0, 10 and 30°C with three repeats per temperature.

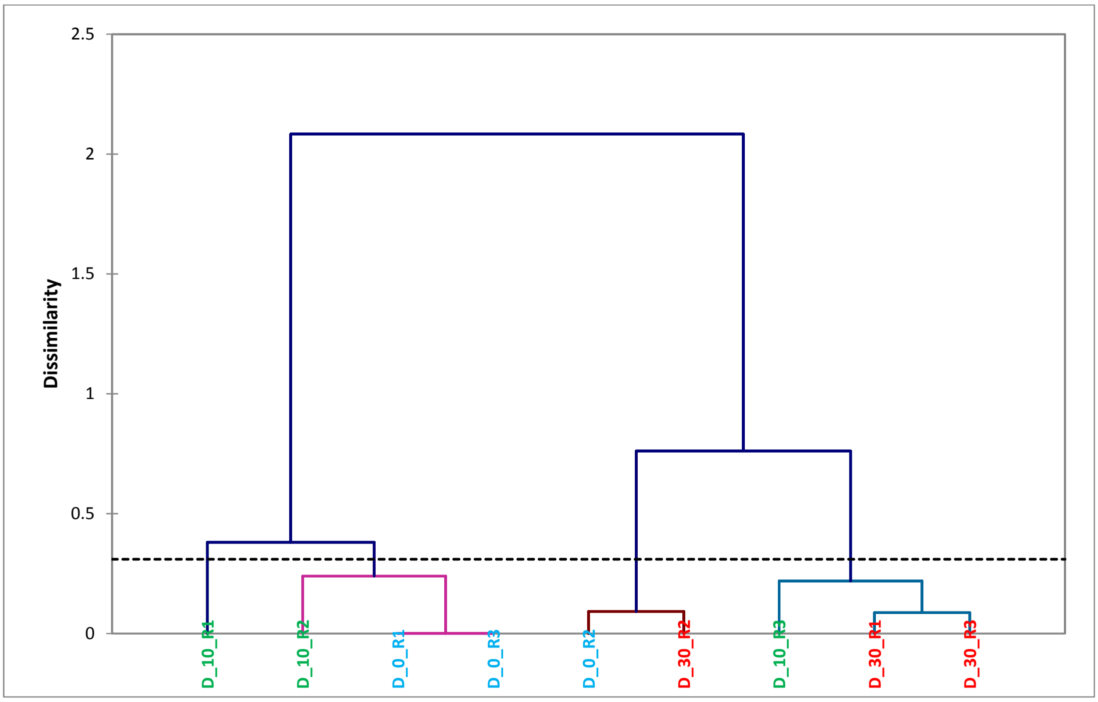

Figure 4.14: Dendrogram of the Agglomerative Hierarchical Clustering (AHC) conducted on 2015 Darling taste sorting of MCCs made from grapes stored at 0, 10 and 30ºC with three biological repeats per treatment.

Table 4.7: 2015 Darling frequency table of taste attributes ranked from highest to lowest. The samples are grouped according to the AHC (Figure 4.14).

	Fruity	**Yeasty/ doughy**	**Fresh**	**Thin body**	**Toasty**	**Medium body**
D_10_R1	6	4	5	3	6	3
D_10_R2	4	5	3	7	1	2
D_0_R1	5	3	3	1	4	3
D_0_R3	6	4	5	3	2	3
Sum	**21**	**16**	**16**	**14**	**13**	**11**
	Yeasty/ doug	**Fresh**	**edium acid**	**Fruity**	**Medium body**	**Slight/ low bitterness**
D_0_R2	6	5	4	3	4	2
D_30_R2	6	5	4	5	3	4
Sum	**12**	**10**	**8**	**8**	**7**	**6**
	Yeasty/ doug	**Fruity**	**edium acid**	**Fresh**	**Bitter**	**Toasty**
D_10_R3	3	4	5	5	4	1
D_30_R1	6	5	3	3	4	2
D_30_R3	5	3	3	3	2	5
Sum	**14**	**12**	**11**	**11**	**10**	**8**

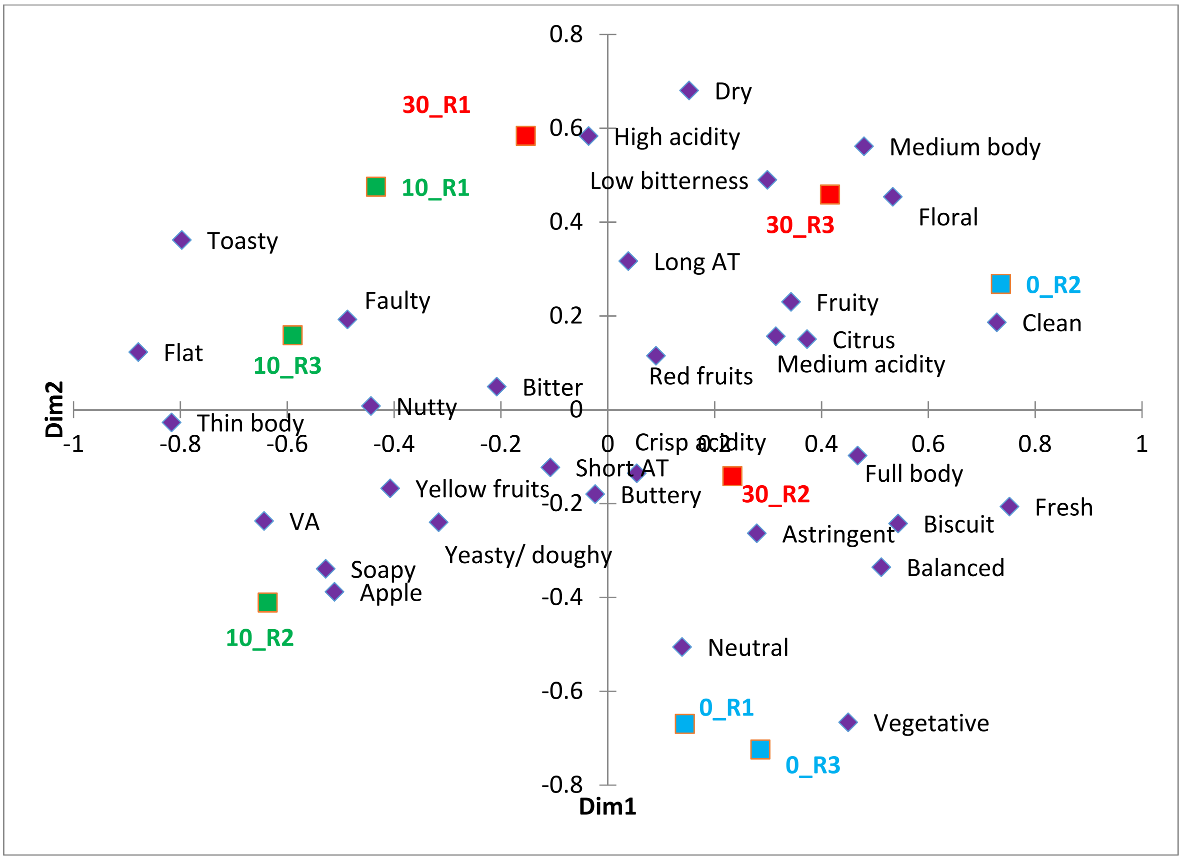

Figure 4.15: Scatter plot of taste sorting results of MCCs made from grapes harvested from Robertson in 2015 and stored at 0, 10 and 30ºC with three repeats per temperature.

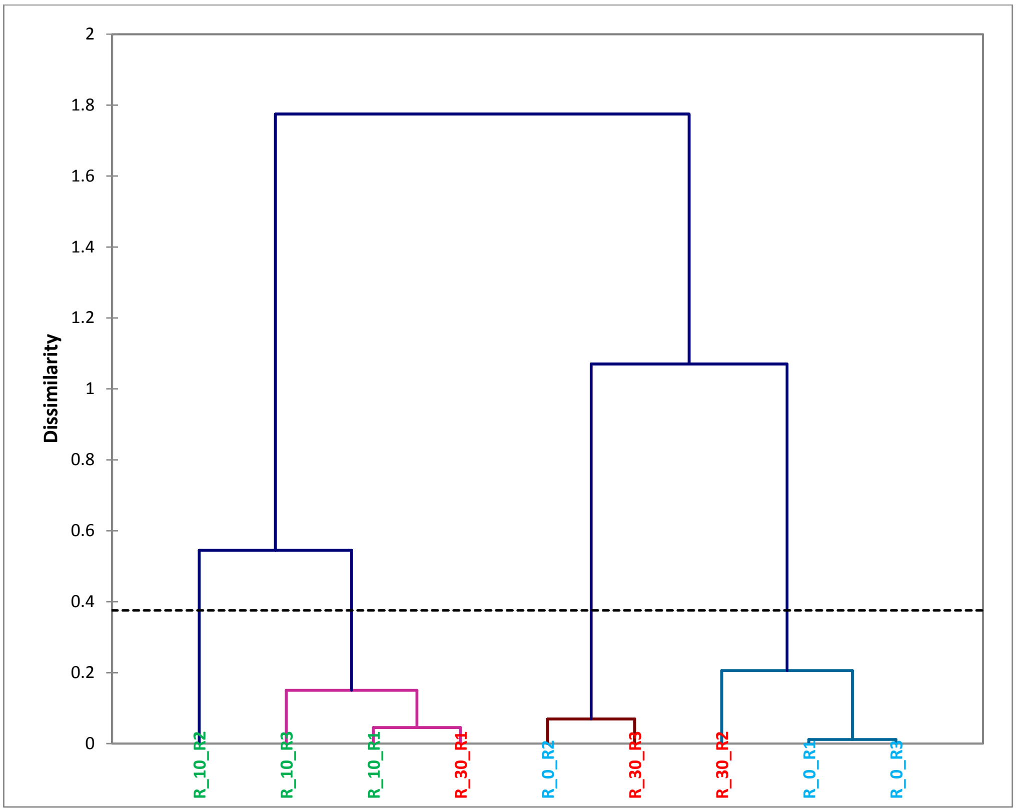

Figure 4.16. Dendrogram of the Agglomerative Hierarchical Clustering (AHC) conducted on 2015 Robertson taste sorting of MCCs made from grapes stored at 0, 10 and 30ºC with three biological repeats per treatment.

Table 4.8: 2015 Robertson frequency table of taste attributes ranked from highest to lowest. The samples are grouped according to the AHC (Figure 4.16).

	Yeasty/ doughy	**Dry**	**Faulty**	**Toasty**	**High acidity**	**Thin body**
R_30_R1	7	5	0	4	3	3
R_10_R1	7	5	4	4	4	2
R_10_R3	6	4	9	4	4	5
Sum	**20**	**14**	**13**	**12**	**11**	**10**
	Yeasty/ doughy	**Fresh**	**Dry**	**Fruity**	**High acidity**	**Buttery**
R_0_R1	6	8	3	2	2	1
R_0_R3	8	5	4	3	2	3
R_30_R2	6	4	4	5	5	4
Sum	**20**	**17**	**11**	**10**	**9**	**8**
	Fresh	**Fruity**	**Medium body**	**High acidity**	**Dry**	**Citrus**
R_30_R3	7	5	6	5	4	5
R_0_R2	6	5	3	3	4	2
Sum	**13**	**10**	**9**	**8**	**8**	**7**
	Fruity	**Thin body**	**Yeasty/ doughy**	**Faulty**	**Fresh**	**Bitter**
R_10_R2	5	5	4	4	3	3

4.4 Conclusion

The storage temperature of grapes had an impact on aroma of the Méthode Cap Classique wines than the taste. Sensory analysis of Cap Classique wines made from grapes stored at 0 and 10ºC showed that the wines had more desirable aroma attributes such as fruity, fresh and floral compared to higher temperature treatments. Grapes stored at 25 and 30ºC produced wines which were associated with positive aroma attributes (i.e. buttery, caramel, oaky and nutty) most commonly associated with sparkling wines but they were plagued with negative aroma attributes such as VA, solvent-like and oxidation, which are not expected of only 9 month-old wines. There were clear vintage differences between the 2014 and 2015 vintages. Sample size also played a role; hence different data sets during the two vintages may have influenced the ability of judges to distinguish between the wines. The 2015 Cap Classique wines had less negative attributes, attributes commonly associated with sparkling wines were cited in all treatments.

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Chapter 5

General discussion and conclusions

5.1 Conclusions and future prospects

Studies on South African wines have covered many important wine related topics from viticulture across winemaking and all the way to consumer perception. This diligence that has been put into the study of different grape cultivars has yet to be put into South African traditional style sparkling wine (TSW) namely, the Méthode Cap Classique wine (MCC).

Wine quality being linked to the phenolic composition of a wine, the current study investigated the phenolic composition of sparkling wines that have been produced from grapes stored at 0, 10, 25 and 30°C to evaluate if the temperature treatments had an influence on the phenolic composition and hence the quality of MCCs. The study used cold rooms commonly used in wineries to store grapes overnight before processing. Furthermore, the study investigated the phenolic composition of these MCCs throughout winemaking to see if there were any changes over time.

In Chapter 3 it was shown that the grape storage temperature has an effect on the phenolic composition of MCCs. Grapes stored at 0 and 10°C produced MCCs with lower total phenolics (TP), colour intensity (CI) and total hydroxycinnamates (TH) compared to MCCs produced from grapes stored at 25 and 30°C. Phenolic concentrations fluctuated slightly across the stages of winemaking but no statistical differences were observed between the samples at different stages of winemaking. This stability of the measured phenolics across winemaking has previously been reported in literature. The study also found phenolic concentrations lower than those reported in literature.

A control experiment where the grapes were not treated would have helped to better understand the effect of the treatment relative to a “real life” system. A possible future study could also look at the impact of the level of phenolics on the perception of MCC wines, in which the level can be increased either by winemaking practices or by in-cellar additions.

Many sensory evaluation techniques have been used for the evaluation of still wines with the aim of gaining statistically solid results that overcome hedonic preferences. In order to find any differences/similarities in these MCCs, a sorting exercise was performed on the aroma and the taste of the MCCs in two separate flights in order to fight sensorial fatigue by the judges. During both the 2014 and 2015 vintages, judges were able to distinguish the aroma of the wines according to the treatments, grouping lower temperature treatments together and higher temperature treatments together. Lower temperature treatments produced MCC wines with positive aroma attributes (ex. *fresh, fruity, citrus*). Higher temperature treatments produced MCC wines, which were associated with negative attributes (ex. *oxidation, solvent-like, chemical*) more frequently cited than the positive aroma attributes. The positive aroma attributes cited for higher temperature treatments were characteristic of traditional style sparkling wines (ex. *yeasty, creamy, autolytic character*). Judges were able to distinguish the wines based on their aroma but not taste, indicating the grape storage temperature had a greater effect on the aroma than the taste/ mouthfeel of the MCCs. Since in our results the treatments affected only the aroma profile of the MCC wines, it would be worthwhile in future to investigate their volatile composition. Using a comprehensive list of attributes rather than free description should be used. Other characteristics, such as effervescence, bubble formation, and colour could also be assessed in a future project, with the help of appropriate sensory methods and physical measurements. Correlations between chemical/physical measurements and sensory could also be investigated if appropriate methods are used.

The project demonstrated that grape temperature at pressing affects the aroma of MCC wines, so the winemaker can make a more informed decision on whether to chill the grapes or not before processing without compromising the taste/ mouthfeel of the wines. The investment in cooling rooms can be costly but offers the possibility of improving on the aroma of MCC wines. Since the sensory evaluation of the two farms investigated (Robertson and Darling) showed slightly different aroma profiles, it would be beneficial to investigate more farms from different climatic regions to see if the patterns observed are maintained.

Appendix A: Vinification and Oenological parameters

Table A1: Oenological data of 2014 Robertson and Darling blends, wines after second fermentation (T2M) and the final wines aged for nine months (T9M) samples.

	Blends				T2M				T9M			
Darling	**0ºC**	**10ºC**	**25ºC**	**30ºC**	**0ºC**	**10ºC**	**25ºC**	**30ºC**	**0ºC**	**10ºC**	**25ºC**	**30ºC**
pH	2.79i	2.82i	2.97efg	2.95fgh	2.87hi	2.91gh	3.14c	3.04de	3.03def	3.10cd	3.35a	3.24b
TA	11.87ab	10.49bc	10.08bc	9.25c	12.02ab	12.63a	10.14bc	11.93ab	11.88ab	10.50abc	9.46c	9.98bc
VA	0.20d	0.24cd	0.26cd	0.40ab	0.24cd	0.41ab	0.46ab	0.44ab	0.23cd	0.28cd	0.49a	0.34bc
RS	2.47a	2.30a	2.23ab	2.30a	1.62cd	1.66cd	1.89bc	1.69c	1.02e	1.06e	1.32de	1.24e
SO_2 (total)	68a	33cd	44b	35c	66a	34c	25d	34c	35c	34c	35c	35c
SO_2 (free)	9b	4de	3e	5cde	6c	6cd	9b	6c	12a	13a	14a	13a
Alcohol	9.89de	9.49e	9.00f	9.04f		10.83a	10.37cd	10.37bc	10.60ab	10.05cd	10.01cd	9.68bc
Robertson	**0ºC**	**10ºC**	**25ºC**	**30ºC**	**0ºC**	**10ºC**	**25ºC**	**30ºC**	**0ºC**	**10ºC**	**25ºC**	**30ºC**
pH	2.89def	2.82ef	3.00cde	3.01cde	2.75f	2.94ed	2.99cde	3.06bcd	3.21ab	3.14abc	3.29a	3.33a
TA	8.13bcd	8.22bcd	7.29d	7.23d	9.56a	9.55a	8.91ab	8.62abc	9.41a	9.62a	7.70cd	7.51d
VA	0.27cde	0.20f	0.23def	0.21ef	0.28cde	0.26def	0.29bcd	0.35ab	0.41a	0.38a	0.34abc	0.35ab
RS	1.66bc	1.50bc	1.74bc	2.05bc	1.33bc	1.45bc	1.66bc	2.60b	1.12c	1.10c	2.43bc	4.43a
SO_2 (total)	36abc	35abc	31bc	28c	40a	38ab	30c	29c	31bc	36abc	33abc	38ab
SO_2 (free)	8d	8cd	7d	8d	10cd	10cd	9cd	9cd	14ab	14ab	11bc	15a
Alcohol	10.93bc	10.78c	10.45c	10.70c	12.18a	12.07a	11.60ab	12.01a	11.65a	11.54ab	11.90a	11.98a

Note: The following are averages over the triplicates with statistical differences calculated at $p<0.5$ across treatments and winemaking stages. TSO_2 - total sulphur dioxide; FSO_2 - free sulphur dioxide; TA - titratable acidity.

Table A2: Oenological data of 2015 Robertson and Darling blends, wines after second fermentation (T2M) and the final wines aged for nine months (T9M) samples.

	Blends				**T2M**				**T9M**			
Robertson	**0ºC**	**10ºC**	**25ºC**	**30ºC**	**0ºC**	**10ºC**	**25ºC**	**30ºC**	**0ºC**	**10ºC**	**25ºC**	**30ºC**
pH	3.18cd	3.29c	3.24cd	3.31c	3.60ab	3.63ab	3.41bc	3.05d	3.69a	3.75a	3.34c	3.26cd
TA	7.62bc	7.21bc	8.45ab	7.16bc	7.78bc	7.45bc	9.17a	8.18 ab	7.52bc	6.47c	7.67bc	7.19bc
VA	0.44ed	0.57cd	0.76ab	0.51ed	0.22f	0.43e	0.64bc	0.20fg	0.08g	0.26f	0.83a	0.28f
RS	1.04cd	1.22cd	2.15abc	1.50cd	2.03bcd	2.87ab	3.20a	1.03d	1.15cd	1.07cd	1.01d	1.07cd
TSO2	33bcd	29cde	38ab	31cde	42a	37abc	34abcd	25de	33bcd	23e	32bcde	28de
FSO2	15a	8ef	7fg	8fg	11cd	12bc	9def	6g	12bc	13ab	10de	8ef
Alcohol	11.71cd	11.65de	10.98g	11.33ef	12.11b	11.84bcd	11.11fg	10.98g	12.47a	12.07b	11.30fg	12.00bc
Darling	**0ºC**	**10ºC**		**30ºC**	**0ºC**	**10ºC**		**30ºC**	**0ºC**	**10ºC**		**30ºC**
pH	3.34d	3.48cd		3.45cd	3.56bc	3.69ab		3.72a	3.57bc	3.66ab		3.78a
TA	9.16ab	8.33c		8.62bc	9.56a	8.70bc		9.07ab	9.21ab	8.76bc		7.03d
VA	0.42d	0.54c		0.73a	0.46cd	0.48cd		0.65b	0.13e	0.16e		0.66ab
RS	1.05d	1.07d		1.40cd	2.63b	3.77a		3.00b	1.40cd	1.15cd		1.73c
TSO2	16bcd	14d		12d	21ab	23a		14d	20abc	22a		14cd
FSO2	6bcd	5de		4e	5de	6cde		5de	7abc	8a		7ab
Alcohol	11.24cd	11.28cd		11.01d	11.48abc	11.37bc		11.27cd	11.70ab	11.70a		11.53abc

Note: The following are averages over the triplicates with statistical differences calculated at $p<0.5$ across treatments and winemaking stages. TSO_2 - total sulphur dioxide; FSO_2 - free sulphur dioxide; TA - titratable acidity.

Appendix B: Sensory evaluation

Judge ___________ **Session** __________ **Date** ______________

Sorting task

You have in front of you 9 samples. We ask you to **smell/ taste** the samples and to group them according to their similarity. Similar samples should be put together and different samples should be in different groups. You can make as many groups as you want and put as many wines as you want in one group (but more than 1 group and less than 9). Once you have sorted the groups, please write down the words that best describe each group.

Please choose the descriptors from the list given. If you find some other attribute not included in the list that you would like to report, please feel free to do so.

You can take as much time as you want.
Thank for your participation.

Write your groups of the products below:

IMPORTANT
Please double check that you have used all 9 sample codes.
Each code can be used only once.

Figure B1: Sorting analysis instruction sheet provided to judges for the aroma sorting exercise (note: the taste instruction sheet only differed in the highlighted “smell” to “taste”.).

AROMATIC DESCRIPTORS LIST

FRUITY
WHITE FRUITS
Pear
Fresh Apple
Yellow Apple
Green Apple
Oxidized Apple

YELLOW FRUITS

CITRUS

RED FRUITS

FRUITY

NUTTY

TROPICAL FRUITS

SWEET ASSOCIATED
Baked Apple
Ripe Fruit
Marmelade

VEGETATIVE / GREEN
VEGETATIVE
FRESH
Herbaceous

SPICY

FLORAL

TOASTED / WOOD
TOASTED
Biscuit
Caramel
Toated Bread
Vanilla

WOODY
Toasted Oak

OTHER
Mineral / Flinty
Buttery
Yeasty / doughy
Autolytic character
Low Lees
Medium Lees
High Lees

VA
Solvent like
Oxidation
Faulty

Sulphur Related

Animal

Aged

FOREST FLOOR
Mouldy

TASTE DESCRIPTORS LIST

Fresh
Clean
Neutral
Off Dry
Fruity
Yeasty
Buttery
Vegetative
Toasty

Faulty
Soapy
Dry

Medium Sweet
High Sweet

Crisp Acidity
High Acidity

Low Bitter
Bitter

Balanced
Well developed
Astringent

Thin body
Low Body
Medium Body
Full Body
Medium Acidity

Short after taste
Long after taste

Figure B2: Attributes sheet provided to judges for the 2015 sorting exercise compiled from 2014 sorting results.